

National Bureau of Standards



December 1959
Volume 43
Number 12

Technical News Bulletin

Special Issue—

Standards and Calibration Activities in Rapid Growth

Industry and Military Share in Recent Upswing

STANDARDS and calibration activity by private industry and the military services has registered a significant increase in recent months and is rapidly gaining in momentum. Led by "space-age" industries, but strongly seconded by other fields such as computers, microwave components, and solid-state electronics, the expansion of activity has taken the form of: (1) Surveys by industry and the military of present and anticipated calibration requirements and measurement problems; (2) considerable growth in size and numbers of standards laboratories within private industry; and (3) the planning and partial establishment by the military services of more extensive systems of standards laboratories at various echelons between the National Bureau of Standards and the weapons, radar, communications, and transport systems at the point of use.

A summary of some of these developments is given in the present article. Other articles in this issue of the *Technical News Bulletin* deal primarily with work being done at the Bureau to meet the demands for improved standards and measurement techniques that are among the more challenging consequences of the upswing in calibration activity.

Studies by Defense Agencies

Among the more unmistakable symptoms of accelerated activity in the standards and calibration field are the numerous studies and surveys it has stimulated. These have attempted to clarify the nature of the new needs that are arising and to canvass the remedial actions that might be taken—expansion of calibration facilities, development of improved standards or precision measuring equipment, unification or other simplifications in the administration of standards laboratories, support of training programs, preparation of literature on calibration techniques and dissemination of other state-of-the-art information, and others.

In the military agencies, surveys and studies have paved the way for major expansions of their standards laboratory systems. Thus it was as a result of studies extending from 1954 to 1956 that the expanded calibration program of the Navy Bureau of Ordnance was set going in 1957. At about the same time a survey of the performance of shipborne electronic equipment led the Navy Bureau of Ships to organize POMSEE (Performance, Operational, and Maintenance Standards of Electronic Equipment), a program to insure

periodic measurements of important parameters of electronic equipment.¹

It was in 1957 that the Quality Control Office of the Air Materiel Command made a study that led to the recommendation that precision measurement laboratories be established at major Air Force Bases. "Project Test Ship" was set up at March AF Base, California, to prove the feasibility of this proposal; the project consisted in assigning all calibration and repair of test equipment at March AFB to a central laboratory.² The Air Force plans to have over 160 such Base standards laboratories in operation before very long (see below).

During the past year, the Office of the Secretary of Defense, Research and Engineering, conducted a study with the aim of promoting research and development on measurement problems. Information was drawn from sources in all the Services and was summarized as a list of measurement techniques and devices whose development was necessary to provide new or more accurate or faster calibration services for defense activities. The list was recently transmitted to the Bureau, and is now being used for guidance in charting the NBS research program.

Industry Survey

In industry, the outstanding survey was in the form of a questionnaire circulated to member companies of the Aerospace Industries Association (AIA). Suggested originally by the Air Materiel Command, the idea of a calibration and standards questionnaire was adopted unanimously in November 1958 by the AIA Quality Control Committee, which proceeded—with some technical advice from NBS—to prepare a questionnaire in two parts. Part I dealt with general problems such as traceability of standards to the Bureau and needs for training and information; Part II inquired in considerable detail about measurement problems in about 200 specific areas.

A general summary of the replies received from 68 AIA member companies was made public on August 25; and the total number of replies received has since reached 79. Of the 68 replies summarized in the report issued in August, 29 were from companies or company subdivisions having 5,000 or more employees

and 36 were from organizations with 500 to 5,000 employees. The results are thus more than adequately representative of the principal airplane, space, and missile companies that constitute the AIA.

The results on Part I of the questionnaire showed, among other things, that most companies were not able to trace their calibrations clearly back to the national standards, and that there was a widely felt

The Measurement Challenge . . .

THE National Bureau of Standards is charged with the responsibility of establishing and maintaining the primary standards of physical measurement of the United States. This responsibility carries with it the mission of providing the central basis for a complete consistent system of physical measurement of national scope adequate for the expanding national activity in scientific research and industrial technology. Consistent with its responsibility and mission, the Bureau conducts a broad research program to advance the science of measurement, to develop new standards, to refine and improve existing standards, to develop improved instrumentation, to develop reliable and objective methods of test, and to provide means for transferring measurement accuracy to scientific and technological organizations.

The demand for the Bureau's services has grown substantially and rapidly in the past few years. This is due to a burgeoning, energetic technical development in the United States, to rapid entrance into new scientific areas, and to an increasing recognition that scientific exploration and technological production are intimately related to effective national progress. It is vital that the National Bureau of Standards maintain a position of leadership in the measurement sciences in order to satisfy these expanding requirements for its services.

Unfortunately, the total Bureau program in these areas is not sufficient to meet the pressing and growing demand. The Bureau, however, is actively aware of the fundamental significance of these services. It is seeking to meet the present critical situation in three ways: (1) By enlarging and strengthening its own calibration services program; (2) by encouraging industry to undertake improved calibration services within particular areas; and (3) by developing greater cooperation between government and industry in defining and attacking the most important of these problems.

A. V. Astin, *Director, NBS*

THROUGHOUT this special issue of the Technical News Bulletin, reference is made to the Industry Calibration Survey conducted by the Aerospace Industries Association. Requests for information on this survey should be addressed to Rear Admiral Richard M. Oliver, U.S.N. Ret., Secretary, Quality Control Committee, Technical Services, Aerospace Industries Association, 610 Shoreham Building, Washington 5, D.C.

need for training and publications in the field of calibration techniques. Replies to the more technical Part II have supplied concrete instances of measurement problems previously suspected in a general way. However, in order to analyze many of the reported difficulties so that they can provide goals of Bureau research, two further steps are being carried out: (1) Bureau staff members are examining the individual (anonymous) replies to the questionnaire; and (2) supplementary information is being sought through followup inquiries from the companies that submitted replies.

Industry Standards Laboratories

It hardly needs to be pointed out that the Bureau cannot calibrate single-handedly all the measuring equipment in use. To take an extreme example, in order to check ordnance components for conformity with specifications, the U.S. Navy must keep roughly a million dimensional gages in calibration, a function that Navy standards laboratories have been performing for over 20 years;¹ and these laboratories in turn have their top standards calibrated by the National Bureau of Standards. Similarly, many industries submit their master standards to the Bureau for calibration, and then use them to calibrate instruments at the working level. The maintenance of reference standards by private industry—not to mention universities and other institutions that do research in physical science and technology—is therefore not a new development.

However, a number of factors now appear to be leading industry to put its standards operations on a broader and more systematic basis. Among these factors are: (1) The need for more accurate data on which to base the engineering design of complex equipment; (2) the need for more accurate monitoring of manufacturing processes to assure realization of the reliability potential of the given design; (3) the greatly increased measurement and calibration workload; and (4) the extension of measurement needs to new areas and to extreme values—for example, to very large forces (rocket thrust) or to very high and very low frequencies, temperatures, and pressures. To solve these problems requires a battery of modern high-accuracy apparatus manned by a staff with the professional knowledge and techniques needed to exploit the capabilities of the available equipment and with the understanding needed to keep up with or even contribute to advances in the state of the art.²

When it became evident earlier this year that there

was a speedup in the establishment of new standards laboratories (or the expansion of old ones) in industry, the Bureau announced its willingness to assist those concerned in every way it could;⁴ this announcement was, in fact, no more than a restatement of established Bureau practice. Since then the Bureau has been host to a stream of visitors who come to spend a day—sometimes two or three—in a round of conferences with staff members, seeking advice on calibration methods, on the characteristics of different types of standards and auxiliary apparatus, and on the design of laboratory buildings, layout, and environmental controls.

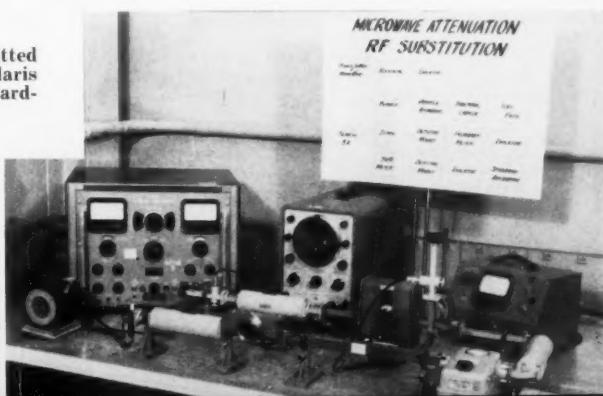
The following summary statements, based on information derived from visitors during just the last few months, may indicate the nature of what is happening: (1) Three corporations have set up standards laboratories in each of at least five of its plants and are planning "corporate-level" standards laboratories to service the plant laboratories; (2) another large company is resurveying its measurement needs in various areas and studying the "desirability and feasibility" of establishing a centralized and expanded corporate-level laboratory; (3) two major automotive firms are expanding their standards laboratories to handle a wider range of measurement areas; (4) a microwave components plant, subsidiary of a large electrical company, was setting up a standards and calibration section; and (5) at least half a dozen other companies indicated that they were already setting up new standards laboratories or were well on the way toward doing so.

Visits during the past year by Bureau staff members to defense contractors have brought the additional information that most, if not all, of the larger missile manufacturers have been adding rapidly to their calibration facilities. Some of these are now on an elaborate scale; several manufacturers have said that the main obstacle to further expansion is the shortage of competent personnel. It may also be noted that a Navy Bureau of Ordnance survey showed there were 207 contractors' Standards Laboratories that were either operational or in the planning stage at the start of the present year. These included only Bureau of Ordnance contractors doing high-priority work.⁵

Military Standards Laboratories

The importance of precision measurements in the testing and maintenance of weapons and supporting systems in the national defense establishment has been recognized and calibration programs of greater or lesser scope have been in effect for several decades at

This U.S. Navy submarine tender (below), is being fitted out as a floating calibration laboratory to service Polaris equipped vessels. Some of its equipment for standardizing microwave components is at right.





U.S. Air Force mobile calibration laboratory is flown to its assignments throughout the country.

least. However, developments during the past year have been on a scale not previously attained, and the indications are that the present rate of expansion will continue for some time.

The more spectacular changes in the immediate past have occurred in the Navy and Air Force; and the Army has been far from inactive—it has a calibration program of substantial proportions and is currently restudying its calibration needs to determine whether further expansion is called for. Also important in the military calibration picture is the well-developed program that has grown up under the Atomic Energy Commission to handle the measurement needs of nuclear weapons systems.⁶

In the military standards laboratory systems sketched below, attention is given to a broad range of measurement areas, including electrical and electronic, dimensional and mechanical, thermal, and radiological. Though electrical and electronic measurements account for the largest part of the total workload, the critical importance of the other areas is well recognized. Thus, measurements of high pressures and forces and of extreme temperatures (high and low) must be made in testing rocket engines; dimensional measurements are as indispensable for the conventional job of assuring that shells will fit the bores of rifles as for the more glamorous task of adjusting highly sensitive inertial guidance devices; and the need to calibrate the "Radiac" devices used for radiation measurements is one to which all the Services have recently been giving greater attention.

Army calibration activities are the responsibility primarily of its Ordnance and Signal Corps. The Army Ordnance Corps is concerned with production phases of weapons as well as with their use in the field. It has three top-level standards laboratories, including the one that has serviced Army ballistic missile work at

Redstone Arsenal, Alabama; two Secondary Reference Laboratories; and 27 calibration centers in the field and at contractors' plants. The Signal Corps, with its responsibility for communication and radar, is well aware of the problems of monitoring electrical and electronic measurements; and it operates a system of standards laboratories that serve calibration needs at various levels—research, manufacturing, field test, and maintenance. Its Field Calibration Service⁶ makes use of large, modified buses as mobile calibration facilities; six such vehicles were completed in August 1957, one was sent to the Army Command in Europe last year, and more recently several more were put in service at Signal Depots in continental U.S.

The Navy,⁷ like the Army, has two principal calibration programs, one in the Bureaus of Ordnance and Ships, the other in the Bureau of Aeronautics. At the top of the BuOrd-BuShips calibration structure are two Primary Standards Laboratories at Washington, D.C., and Pomona, California, that service the east and west coasts, respectively; these have been gathering equipment and staff during the past year and are now essentially complete. At the next echelon are 10 BuOrd Secondary Standards Laboratories and 12 BuShips Reference Laboratories; staff and equipment for these are expected to be nearly complete by the end of 1959 and they should become operational in the course of 1960. Then there are a larger number of local standards laboratories that are in the charge of contractors or of inspectors at BuOrd or BuShips field activities. BuShips is also planning a fleet of "floating standards laboratories" which will be set up in Navy tenders; eight of these are expected to be operational by 1960 and a considerably larger number are being planned.

The Navy BuAer calibration program makes use of four Primary Standards Laboratories, all of which were considerably expanded in the course of the past year and are still growing; they are located at the Naval Air

U.S. Army Signal Corps field calibration unit. At right is the interior of the vehicle.



stations at Alameda, Pensacola, Norfolk, and North Island (California). Below these laboratories are 12 Reference Standards Laboratories, including two overseas, that bear the brunt of the program; they are responsible for calibrating all production test equipment located at their parent Naval Air Stations and all fleet "avionics" test equipment in their particular geographical areas. In addition, many smaller facilities are maintained to calibrate acceptance test equipment at contractors' plants.

The Air Force has been moving rapidly in the past year toward a single, integrated calibration system, administered for the Air Force as a whole by the Quality Control Office of the Air Materiel Command. At the top of the chain of laboratories that constitute the system is the USAF Calibration Laboratory at Dayton Air Force Depot.² In the nonelectrical areas, this laboratory maintains the AF standards that are used to calibrate the standards at the next lower echelon, the "AMA-level" laboratories which are located at various Air Materiel Areas (AMA's) and certain AF Depots. For the electrical and electronic areas, the Dayton laboratory has a detachment at the NBS Boulder Laboratories which maintains a pool of Air Force standards; these are calibrated by the NBS Electronic Calibration Center and are exchanged on a regular schedule for the standards in use at the AMA-level laboratories. At present there are 16 AMA-level laboratories, of which 11 are in continental U.S. (including Alaska), two in Canada, and three overseas. Several of these have received only a very limited set of standards and equipment thus far; and many of the others were equipped in the course of the past year.

Next below the AMA-level laboratories are the "Base" Standards Laboratories that will normally be located at Air Force Base calibration and maintenance shops. More than 160 such laboratories are now planned; about 40 are expected to be operational by the end of 1959, and the remainder should receive their complements of equipment within the next year.

The highest-echelon laboratories in each of these calibration programs will have their standards calibrated by the National Bureau of Standards; and the top service laboratories will calibrate the standards of the laboratories in the next lower echelon, and so on down the line. However, there is a certain loss in accuracy (the amount depending on the measurement area) in passing from one level to another in the calibration hierarchy, so that it is often necessary to skip some of the steps in the normal chain of calibrations. Thus, many of the standards being placed aboard the Navy tenders will need to be calibrated directly against the top Navy standards or even against those at the Bureau; and as another example, the force measuring devices used to determine thrust in routine rocket tests are being sent in increasing numbers directly to NBS in order to realize the maximum attainable accuracy.

To some extent the calibration programs briefly sketched above have overlapping functions, a fact of which the Services are aware, and interservice studies are now being made to determine ways of reducing it to a minimum. In a number of geographical areas,

Measurement of Small Internal Diameters

A Correction

THE BUREAU is now equipped to measure internal diameters as small as 0.040 in. with an accuracy of about 10 μ in. In the tabular report of a survey conducted by the Aerospace Industries Association,* a note regarding this measurement was inadvertently placed in the wrong column and led to the erroneous impression that the accuracy of the Bureau's measurement capability for internal diameters was only 0.040 in.

**Aerospace Industries Association, Industry Calibration Survey Part II, page 6.*

cross-servicing agreements are already in force by which one of the military agencies undertakes to provide calibration services to another agency. Efforts are also being made to coordinate techniques and practices and to see that printed calibration procedures prepared by any one of the services are made available to the others.

¹ BuOrd-BuShips calibration program, by Lt. F. L. Roach, *IRE Trans. Instr.*, Vol. 1-7, Numbers 3 and 4 (double issue), p. 357 (December 1958).

² USAF program for calibration and repair of precision measurement equipment, by Major K. M. Johnson, *U.S. Air Force Aircraft Accident and Maintenance Rev.* 14, 2 (October 1959).

³ Accounts of the development and facilities of three major industry standards laboratories, as of the end of 1958, are given in the following articles of the *IRE* publication referred to in footnote 1: The organization, administration, and operation of an industrial standards laboratory, by J. N. Whitaker, p. 345; A centralized facility for electrical and microwave calibrations in a large company, by L. B. Wilson, p. 348; and Evolution of a standards laboratory, by C. E. White, p. 339.

⁴ Assistance to other standards laboratories, *NBS Tech. News Bul.* 43, 21 (February 1959).

⁵ Communication from the Metrology Department, Naval Inspector of Ordnance, Pomona, California. It should be noted that the contractors' laboratories varied greatly in the level of accuracy at which they operated and in the scope of their measurement activities—some covering only a few areas, others covering an extensive range—depending on the type of work done by the individual contractor.

⁶ Electronics communications procedure: Field Calibration Service, a pamphlet prepared by the Sacramento Signal Depot for the U.S. Army Signal Corps (September 1957); and communications from the Office of the Chief Signal Officer.

⁷ The BuOrd-BuShips calibration program is described in the reference given in footnote 1; see also The Navy's test equipment calibration program, by Lieutenant Commander L. O. Whaley, U.S.N., in the *Bureau of Ships Journal* (January 1959). The BuAer program is described in Instrument calibration program with the Department of the Navy—Bureau of Aeronautics phase, by M. L. Scroggs, p. 360 of the *IRE* publication referred to in footnote 1.

⁸ See the account given in the *IRE* publication referred to in footnote 1: Physical and electrical standardization program for the AEC, by H. C. Biggs, p. 364.



STANDARDS AND CALIBRATION IN RADIO AND ELECTRONICS

ELectronics is now the fifth largest industry in this country. It has been predicted that it will step into first place within 10 years. With this rapid growth has come the problem of accurately measuring the basic radio and electronic quantities so as to insure the unfailing performance of an ever-increasing variety of components and equipment.

Such frontier areas as space exploration, automation, and miniaturization are subjecting electronic equipment to new and complex uses as well as extreme environments. In these applications the need for accuracy on the production line becomes increasingly important. In the missile field, for example, it is estimated that reliability above 90 percent can be achieved only if each component has not more than a 1-to-1,000 probability of failure. To produce components having the necessary uniformity and accuracy requires a chain of calibration leading from the assembly line back ultimately to the precise electrical standards maintained for American science and industry by the National Bureau of Standards.

As indicated by the recent Aerospace Industries Association Survey,¹ the precision needs of the military services and industry are now outstripping the avail-

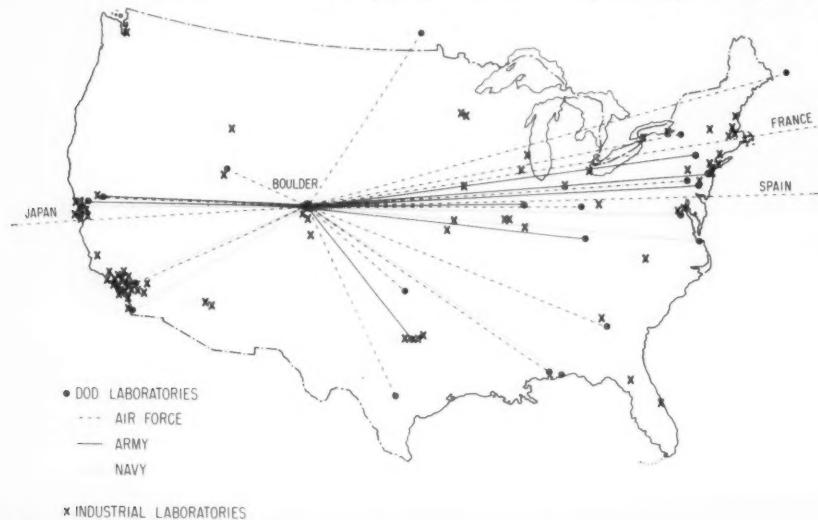
Charles Snider pours liquid nitrogen into a cold trap of the new cesium-beam frequency standard as Roger Beehler adjusts the atomic beam detector. The U-shaped resonant cavity used to excite the cesium transition is suspended above the center of the machine.

ability of standards and calibration services in the radio-electronics field. Although manufacturers have attempted to fill the gap by establishing procedures to calibrate their own working standards, these working standards lose much of their value if they are not calibrated in terms of the national standards.

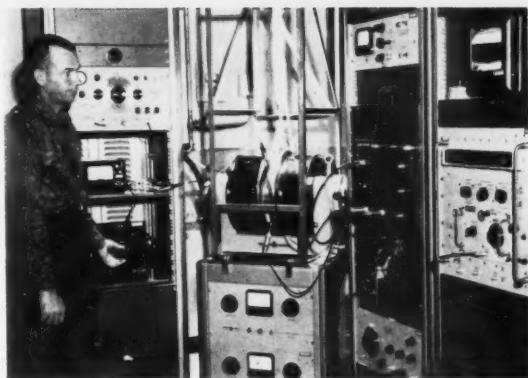
In an effort to meet these urgent needs, the Bureau's Radio Standards Laboratory in Boulder, Colo., is expanding its program of radio standards research and calibration services. Within the limits of its facilities and staff, the Laboratory is seeking to provide the improved standards, measurement techniques, and associated instrumentation that are needed for all radio frequency and microwave quantities. At the present time, standards are being established or improved for frequency, power, attenuation, voltage, impedance, noise, field strength, interference, conductivity, dielectrics, and magnetics. In addition, a new laboratory has recently been activated to study radio properties of materials with advanced techniques.

Microwave Impedance

Impedance measurements play an essential role in the design, production, and evaluation of electronic equipment; however, it is also one of the most difficult



Top military and industrial reference laboratories served by the NBS Electronic Calibration Center during its first year of operation. Most of these in turn calibrate lower-level standards.



William Case adjusts the local oscillator in an experimental setup for evaluating the tensor permeability properties of ferrite materials. The ferrite (in cavity of electromagnet) is measured at 1,060 Mc.

quantities to measure accurately. Recently the Radio Standards Laboratory significantly improved impedance standards and measurement techniques in the microwave range.

Three types of fixed impedance (or reflection) standards have been developed: An adjustable sliding termination for rectangular waveguide, which can be arranged to have practically no reflection; very precise short-circuited sections having almost total reflection; and half-round obstacles whose reflections can be calculated from the dimensions and wavelengths.

The adjustable sliding termination has a voltage standing-wave ratio of less than 1.0002 or a return loss greater than 80 db. Extremely fine mechanical tolerances and controls provide a fine adjustment and minimum variation in reflection.

In the short-circuited sections of waveguide the input flange is a quarter wavelength from the short circuit. In a typical example a short-circuited section of X-band electroformed silver waveguide has a calculated VSWR at 10 kMc of approximately 5,140, corresponding to a voltage reflection coefficient of approximately 0.99961.

To test these short-circuited sections it was necessary to know the effective conductivity of the metal. This conductivity was obtained by making attenuation measurements of the sections. In these measurements, an attenuator was calibrated by modifying a system used in microwave power research. The lower range of the microwave variable attenuator was calibrated at approximately 9.4 kMc to accuracies exceeding 0.0001 db. Such accuracy exceeds that to which fine attenuators can be read. This development illustrates the interdependence of basic measurements. In this case, the need to evaluate impedance standards revealed a need for attenuation measurements that was met by a modification of a power measurement system.

From a theoretical analysis, inductive half-round obstacles have been built for use as impedance standards over a wide range of reflections. Measurements of these reflections have agreed with calculated values to better than 0.1 percent in VSWR.

The calibration and use of these standards required

improvement in the measurement of microwave impedance. Accuracies of 0.1 percent in VSWR to 2.0 were achieved by using magnified response and modified reflectometer techniques. The development of the latter technique included a rigorous analysis of the microwave reflectometer. This analysis describes the correct adjustment of auxiliary tuners, and provides quantitative values for errors resulting from incorrect adjustments. Work is in progress on the extension of these impedance measuring techniques to other sizes of rectangular waveguide and to coaxial systems.

The above description of recent research and development in microwave impedance illustrates advances in a specific area of standardization. Progress is also being achieved in other basic quantities throughout the radiofrequency range.

Atomic Frequency Standards

The physical quantity most important to the electronic field is frequency. To make the national standards of frequency and time intervals readily available, radio broadcasts are made continuously from WWV, in Beltsville, Maryland, and WWVH in Maui, Hawaii. In addition, a 60-ke experimental station broadcasts from Boulder, Colo.

The Radio Standards Laboratory monitors WWV continuously. Its frequency is measured daily in terms of extremely accurate atomic standards. With recent improvements in technique, comparisons can now be made to a part in 100 billion.

Experiments in the search for more accurate standards of time and frequency have shown that standards based on unvarying properties of atoms are more precise than astronomical or quartz crystal standards. Atomic standards are also simpler and more completely understood. They do not have the secular variations inherent in astronomical time, nor do they suffer from the aging effects of quartz. In addition, they measure time and frequency very quickly, in contrast to delays of months or years necessary for evaluation of other systems.

Recent improvements in atomic frequency standards are opening up new possibilities in science and engineering. For example, atomic clocks provide high-resolution spectroscopic techniques that can be used to probe deeper into the molecule, atom, and nucleus. Also, more accurate time measurement will permit a closer study of the effect of land tides, sea tides, and the motion of air masses upon the rotation of the earth. It may even provide a means of detecting the effect of rarified gases and magnetic fields on the motion of planets or satellites. Another government agency is now planning to use atomic clocks in an experimental test of the special and general theories of relativity.

The atomic beam frequency standards under development in the Radio Standards Laboratory depend upon the transitions of cesium atoms from one energy state to another. These transitions can occur by the absorption or emission of an electromagnetic wave of a very definite frequency. This frequency is determined by the difference in energy of the two states involved in the transition. For an isolated atom the energy dif-

ference of these states—and consequently the emitted or absorbed frequency—is invariant. Of course, the apparatus used to observe the transition disturbs the atoms, and they can then no longer be considered isolated. However, the atomic beam technique creates the least such disturbance of all current methods. For this reason, it is thought to provide the most accurate frequency standard, although perhaps not the most precise.

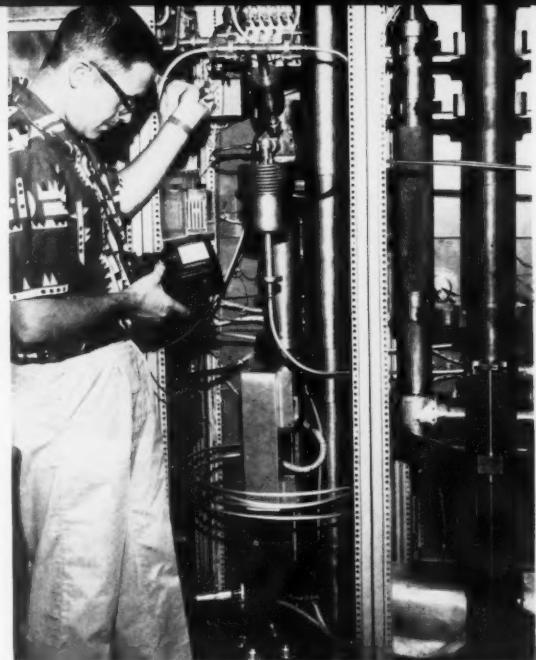
During the past 18 months a cesium beam atomic standard has been almost completely remodeled so that it now has a precision and accuracy of about 7 parts in 100 billion. A new cesium beam standard, designed to have a somewhat higher precision, has just been completed. These two cesium standards, an Atomicon, and an extremely stable crystal oscillator, are now being intercompared on a regular basis.

The new atomic beam is designed to use thallium as well as cesium (thallium has certain important advantages over cesium). Initially, however, cesium is being used and preliminary results (as of October 1, 1959) indicate that its precision and accuracy is 6 parts in 100 billion. The new machine compares in frequency with the old machine to 7 parts in 100 billion.

Two ammonia masers (microwave amplification through stimulated emission of radiation) are being used to study the character of the radiation which excites the cesium transition. At the same time they provide separate standards for frequency comparison. Frequency comparisons with atomic standards in other parts of the world are also made on a regular basis.

Electronic Calibration Center

For quantities other than frequency, the Radio Standards laboratory disseminates its standards and measurement techniques to the Nation mainly through its Electronic Calibration Center,² established in 1953. The primary mission of the Center is to calibrate inter-



David Russell adjusts the "magic T" input to the detector of the new high-frequency piston attenuator (see also photo on facing page). On the right is the large trombone phase shifter used in this dual-channel system.

laboratory standards for such quantities as voltage, power, and impedance in terms of the national standards maintained by NBS. These interlaboratory standards, in turn, are used to assure the accuracy of reference and working standards.

Although the Center was established primarily to meet critical needs within the Department of Defense, it is also designed to meet the needs of the electronics industry. Efforts are underway to increase the capacity of the Center to provide a larger number of individual calibrations each year.

Its calibration activities are divided into three units covering low, high, and microwave frequency measurements. Instrumentation is still incomplete but interim steps are used when necessary to help meet the calibration demand.

Low frequency (zero to 30 kc) instrumentation now provides for the calibration of resistors, bridges, potentiometers, capacitors, inductors, standard cells, electrical instruments, ratio devices, and instrument transformers. Within recent months the Center has received a transformer-type capacitance bridge, constructed by the Electricity and Electronics Division, that will extend the capacitance calibration range, for low frequencies, downward to 1 μ pf. By modifying existing ratio sets and associated equipment, it is expected that the frequency range of current and potential transformer calibrations will be extended, within the year from 60 to 400 cps.



An accurate attenuation measurement technique was developed to evaluate new short-circuit types of microwave impedance standards. Wilbur Anson examines a silver section of waveguide used to transfer this measurement to the impedance standards.



Eugene Amrine uses the new NBS high-frequency piston attenuator to calibrate the unknown inserted in the lower-right panel.

To date, the saturated cells used by the low-frequency unit to maintain the volt have been kept at a reasonably constant temperature in an air bath. A new oil bath that is now being completed should increase certified accuracies five times over the current accuracy of 0.001 percent.

The high-frequency unit (30 kc to 300 Mc) is now equipped to calibrate standards of voltage (unbalanced), power, impedance, attenuation, and field strength. At the present time, these standards are limited to cw measurements. Calibration services for most quantities are at the fixed frequencies of 30, 100, and 300 kc, and 1, 3, 10, 30, 100, and 300 Mc. Continuous frequency coverage is being provided, however, as rapidly as stable and accurate equipment can be devised.

A new precision piston attenuator to operate at 30 Mc has just been completed by the Laboratory. This attenuator will measure a change in attenuation of less than 0.001 db, and will allow calibration accuracies of 0.01 db. These same accuracies are expected to be offered within the next year at 100 and 300 Mc.

High-frequency voltage is now being calibrated at fixed frequencies ranging from 30 kc to 400 Mc. Consoles from 30 kc to 100 Mc cover from 0.2 to 500 v. The 300 and 400 Mc console covers from 0.2 to 100 v. A microvolt calibration console, which is nearly complete, will extend from 1 μ v to 0.1 v, using the fixed frequency sources of the voltmeter consoles. Most of this hf voltage calibration is to an accuracy of 3 percent. The range from 30 kc to 10 Mc, however, is being calibrated to an accuracy of 2 percent, and it is hoped that during the next year this accuracy can be extended to 0.25 percent.

In the microwave range (above 300 Mc) calibration equipment for power, impedance, attenuation, frequency, and noise is being established or improved. Instrumentation using coaxial transmission line components is being prepared for the nominal frequency range 300 to 4,000 Mc, and instrumentation using waveguide components will cover the range of 2.6 to 10 kMc. For frequency measurements, this range is extended to 75 kMc.

Power calibrations in waveguide are now being made to an accuracy of 1 percent in the frequency range of 8.2 to 12.4 kMc. A setup for power measurements in the waveguide range of 2.6 to 3.95 kMc is nearly complete.

Apparatus for microwave impedance (VSWR) measurements in the waveguide range of 8.2 to 12.4 kMc is nearly complete. Initial accuracies are expected to be within at least 0.5 percent and ultimate accuracies may be 0.1 percent.

Microwave attenuation is being calibrated to 0.1 db in waveguide from 2.6 to 18.0 kMc. Equipment for waveguide calibration from 18.0 to 26.5 kMc should be completed during 1960. Attenuation measurements in coaxial line have been extended to 5.6 kMc.

A calibration project to measure microwave noise began in early 1959, and the calibration equipment for waveguide range 8.2 to 12.4 kMc is now almost complete.

During its first year of operation, the Center made 14,182 measurements on the 2,074 items which were calibrated. About two-thirds of these items were for the Department of Defense and about one-third was for the electronics industry.

Radio Materials

The work in measurement and determination of physical quantities at radio frequencies is being extended to measurement of the physical properties of matter with radio techniques. An understanding of the properties of materials at radio and microwave frequencies is important to advances in radio technology where new discoveries often depend on knowledge of the behavior of different substances. For this reason, the Radio Standards Laboratory has established a new facility to study the interactions between electromagnetic waves and matter.

This facility will use the most advanced radio techniques to conduct its research. Molecular beam techniques, radio and microwave spectroscopy, gyrotrons, nuclear resonance, ferromagnetic resonance, paramagnetic resonance, and microwave solid-state amplifiers are among some of the research tools. They will be used to investigate molecular, chemical, and liquid- and solid-state problems; dielectric, magnetic, and semiconductor phenomena; and determinations of new standards and physical constants.

The Laboratory has been studying magnetic phenomena for the Navy Bureau of Ships since 1952. This work began with the evaluation of powdered irons, and has since been expanded to include measurements of

many other magnetic materials under static conditions and at microwave frequencies. For instance, a quasi-static hysteresis loop tester (Cioffi type) is nearing completion and will be used for studies of ferrites. Also a modified vibrating sample magnetometer has been designed to measure the maximum magnetic intensity in a material.

In the range from direct current to microwave frequencies, investigations are underway on magnetostriction (in the rf region only) on the total energy lost by magnetic cores when they are exposed to high-level magnetic fields, and on magnetic and dielectric spectroscopy techniques. Work in the third category includes extensive studies of coils, rf permeameters, and cavities.

Emphasis in the microwave region is being placed on a determination of the tensor permeability (directional properties) and dielectric properties of ferrites at about 1,060, 3,100, and 9,200 Mc. Studies are also being conducted on the tensor permeability of ferrites at low d-c fields. Such information is used to design many new microwave-ferrite devices such as isolators, phase shifters, modulators, and circulators.

Dielectric constants and dissipation factors are measured over a frequency range of 0.1 to 10^{10} cps at tem-

peratures from -100° to $+500^{\circ}$ C. The Bureau has developed methods for measuring loss tangents as low as 0.000,001 at 1 Mc. Facilities for millimeter-wave work are now available, and studies have been made at the low-frequency end of the dielectric spectrum on frequencies so low that their periods are days in length.

A detailed study of polarization and conductivity in crystals of barium titanate has led to formulas that consider the presence of free charges near the domain walls. These equations explain variation in hysteresis loop shape, the dependence of conductivity on polarization, and the variation of switching time with various parameters.

In conductivity studies the Radio Standards Laboratory is investigating the tensor or directional conductivity of semiconductors such as single crystals of germanium at microwave frequencies under different physical conditions. These studies are expected to yield a better understanding of the crystal-lattice forces and processes.

¹ For a detailed report on the Aerospace Industries Association report, see p. 222 of this issue of the Technical News Bulletin.

² For further information, see Services and facilities of the Electronic Calibration Center, *Tech. News Bul.* 223, 42 (Nov. 1958).

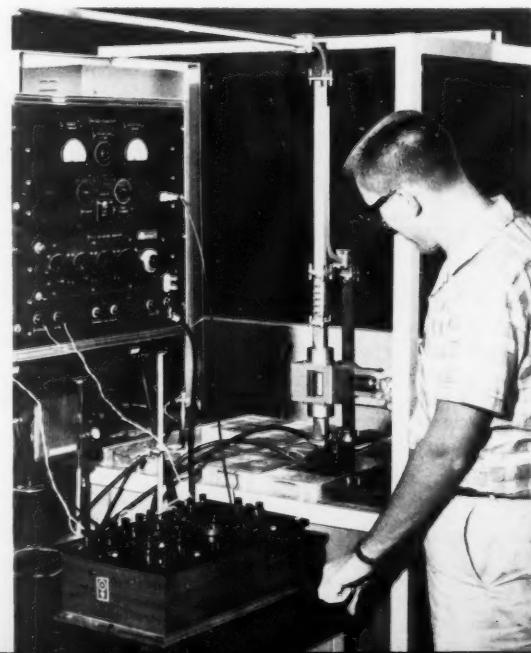
ULTRAPRECISE ATTENUATION MEASUREMENT

A METHOD has been developed for calibrating the lower ranges of a microwave variable attenuator to accuracies better than 0.0001 db (10 microbels). This accuracy exceeds the precision to which available attenuators can be set and read, and is the most accurate measurement of microwave attenuation yet made at the Bureau's Radio Standards Laboratory, Boulder, Colo.

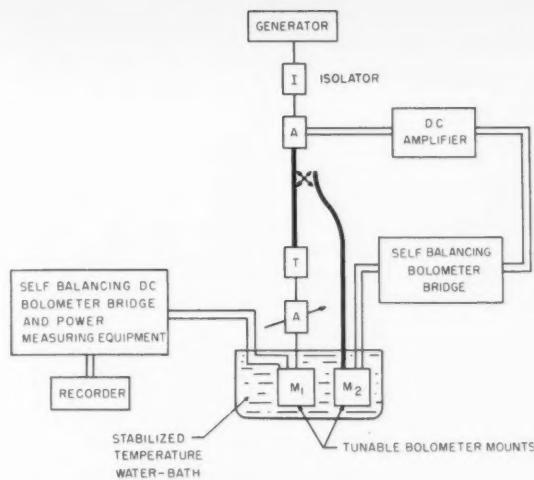
The work was carried out by the personnel of the microwave power standards project under the leadership of G. F. Engen, with assistance from members of the microwave impedance and attenuation projects. This work was done in connection with the Bureau's program to develop microwave standards and precision measurement methods at microwave frequencies. Calibrated microwave attenuators and directional couplers are used in such instruments as field strength meters and signal generators, and in alignment of radar transmitters and receivers. The use of attenuators for power measurements reduces high-power outputs by a known amount to a level that can be conveniently measured with milliwatt instruments. Manufacturers of microwave equipment need their transfer standards calibrated against a national standard to insure the accuracy of attenuators made for industry, military, and the Government. This present development provides the required accuracy in the lower ranges.

The improved accuracy was made possible by adapting a very stable power measurement system to attenu-

tion measurements. The resulting calibration system consists of an amplitude-stabilized microwave signal source and a bolometer detector operated in a temperature-stabilized water bath. There are provisions for "tuning out" the reflections of the system at the place where the test attenuator is inserted, and for accurately measuring the d-c power supplied to the bolometer detector. A second bolometer detector forms part of the amplitude stabilization loop.¹



Measuring attenuation to 10 microbels at microwave frequencies. James E. Gilbert changes the setting of the test attenuator during its calibration.



Calibration system developed at the Bureau for measuring precisely very small attenuations.

The change in attenuation as the attenuator dial is moved from the zero position to some other position changes the microwave power input to bolometer mount from P_1 to P_2 . The measured attenuation is given by the expression $A = 10 \log_{10} \frac{P_1}{P_2}$. The microwave power

as measured by the bolometer technique is proportional to the amount of d-c power withdrawn in order to keep the bolometer resistance constant. The constant of proportionality is the "effective efficiency" of the bolometer mount. This factor cancels out in the expression for attenuation provided that it is independent of power level, and previous experiments have verified this.²

The bolometric measurements are made through use of a self-balancing d-c bridge,³ a constant current gen-

erator, and associated d-c measurement apparatus. This instrumentation provides a direct indication of changes in the microwave power input, which makes possible the determination of small attenuation values.

The attenuation of a rotary vane type of variable attenuator was measured by this method. To obtain an indication of the repeatability of setting the attenuator, the results of three independent settings were recorded. Although the main interest was originally in the lower ranges, the full range of the attenuator was measured.

Two sources of error were considered in estimating the limits of error in the resulting data. These were the mismatch error and the error arising from the uncertainty in measuring the d-c power differences. Calculations of the limits of mismatch error⁴ are based upon system reflections corresponding to a VSWR less than 1.005 and on changes in the attenuator characteristics as determined from separate measurements. The error in measuring d-c power differences is estimated to be less than 0.1 percent $\pm 0.1 \mu\text{w}$.

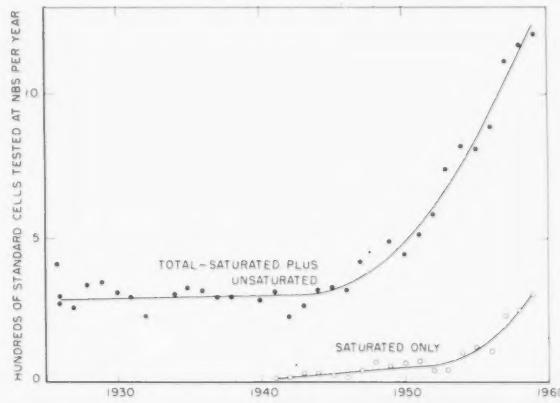
A statistical analysis of the data indicates that the accuracy of the measurements exceeds the precision with which the attenuator can be set and read. This finding suggests that variable microwave attenuators with expanded scales and precise gears are needed if full advantage is to be taken of this calibration accuracy.

¹ Amplitude stabilization of a microwave signal source, by Glenn F. Engen, *IRE Trans. on Microwave Theory and Techniques MTT-6*, no. 2 (April 1958).

² Recent developments in the field of microwave power measurements at the National Bureau of Standards, by Glenn F. Engen, *IRE Trans. on Instr. I-7*, pp. 304-306 (December 1958).

³ A self-balancing d-c bridge for accurate bolometric power measurements, by Glenn F. Engen, *J. Research NBS* **59**, 101-105 (August 1957) RP2776.

⁴ Mismatch errors in the measurements of ultrahigh frequency and microwave variable attenuators, by R. W. Beatty, *J. Research NBS* **52**, 7 (January 1954) RP2465.



The rapid growth in the electrical calibration load at the Bureau is reflected in this graph showing the rise in the number of standard cells calibrated yearly. These cells, which are standards of electromotive force, are tested by comparison with a group of reference standards maintained by the Bureau.

A New Basis for Electrical Standards

BECAUSE of the important role that electricity and electronics play in modern science and industry, the Bureau must work continually to improve the accuracy of electrical measurements, measuring devices, and standards. The latest effort being made in this direction is an attempt to establish an alternate, more accurate foundation for the whole structure of electrical standards. The basis for this new approach is an accurate capacitance measuring bridge and an accompanying calculable standard of capacitance developed last year.¹ This advance allows the electrical quantities of resistance and voltage to be determined by alternate procedures, and provides a check on the ohm and the volt as currently maintained by the Bureau. As all the other electrical units in use today may be derived from these two basic units, it is essential that the ohm and the volt be known as accurately as possible. The Bureau is therefore working to improve the realization of the ohm and has formulated plans for a new realization of the volt.

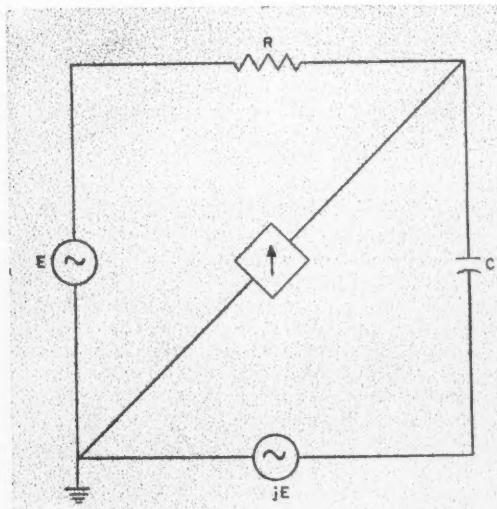
There are many interactions between electrical and mechanical phenomena which offer possible methods for realizing electrical units by means of mechanical measurements. However, until recently only one relation could be exploited with the required accuracy. This is the dependence of the inductance of a circuit on its geometric size and shape. The Bureau has therefore

been using this relation to realize the basic units of electrical measurement—the ohm, the ampere, and, from Ohm's law, the volt. The absolute ohm is based on a computable self or mutual inductor² and the absolute ampere is based on the calculated force between two carefully constructed current-carrying coils.³

The development of the computable cylindrical cross-capacitor and the high-accuracy transformer-type capacitance bridge has changed this situation materially. The computable capacitor can now be used to provide an alternate and probably a more accurate basis for the electrical standards.

The realization of the ohm in terms of the computable capacitor involves comparing a resistive impedance with a capacitive impedance. The capacitance is adjusted until the current through it balances the current through the resistor when the two impedances are connected to voltage sources equal in magnitude but differing in phase by 90 deg. This allows a value to be assigned to the resistor in terms of the known capacitance and the frequency of measurement.

However, the resistors used in maintaining the ohm are not suitable for use with alternating current. A critical part of the new determination is therefore the construction of a comparison resistor which will have the same value at the comparison frequency as when used with direct current. A resistor which is expected to meet this requirement has been designed and is now undergoing final adjustment.



Principle involved in the Bureau's determination of resistance in terms of capacitance. The two voltage sources are equal in magnitude but 90 deg out of phase, and the capacitance is adjusted so that no current flows through the detector. At this balance $R=1/\omega C$, so that the resistance can be determined directly in terms of the capacitance at the frequency of measurement.

R. Cutkosky tests for possible sources of error in the transfer resistor being developed by the Bureau. This resistor will have the same value with alternating current as it has with direct current. It can therefore be compared with a calculable capacitor in an a-c circuit and then used to evaluate the resistors which maintain the ohm. These standard resistors cannot be used directly with alternating current.



A realization of the absolute volt through capacitance measurements is also being considered. This determination, like that of the ampere, will be based on a force measurement. Here the force between plates of the capacitor is proportional to the square of the impressed voltage and to the rate of change of capacitance with displacement. It is expected that measuring this force will provide a realization of the volt with an accuracy at least as high as, and probably higher than, present absolute-ampere determinations.

¹ New capacitance standards, *NBS Tech. News Bul.* **42**, 229 (1958); for further technical details, see A new theorem in electrostatics and its application to calculable standards of capacitance, by A. M. Thompson and D. G. Lampard, *Nature* **177**, 888 (1956).

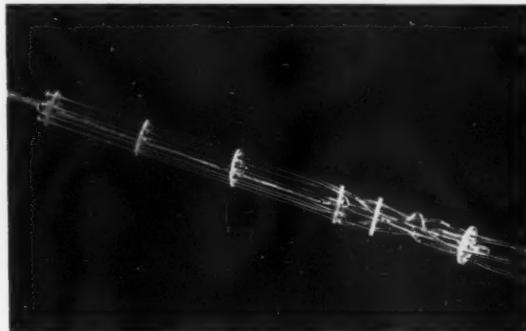
² Standards for electrical measurement, *NBS Tech. News Bul.* **34**, 176 (1950).

³ Redetermination of the standard ampere, *NBS Tech. News Bul.* **42**, 21 (1958).

High-Temperature Resistance Thermometry

THE Bureau has developed a platinum resistance thermometer for interpolating between fixed points on the International Temperature Scale (ITS) above 630.5° C., the melting point of antimony. In addition to simplifying and smoothing the scale, it is expected that its use at temperatures up to 1,063° C (the gold point) will provide greater precision and reproducibility than is possible with present thermometric instruments.

The continual development of new technological processes that occur at high temperatures requires temperature determinations with increasing accuracy and the extension of temperature-measuring techniques to higher temperatures. The International Temperature Scale is presently defined, for the range 630.5° to 1,063° C., in terms of a platinum versus platinum-rhodium thermocouple. This instrument, however, can measure with an accuracy of only a few tenths of a degree. The platinum resistance thermometer is presently used at temperatures below 630.5° C.; extension of its range to 1,063° C., to replace the thermocouple, would not only provide greater precision, but would also simplify and smooth the scale. Moreover, development of a stable high-temperature resistance thermometer will be helpful to precision gas thermometry at temperatures above 630.5° C.



Temperature sensing resistor of platinum resistance thermometer developed at the Bureau for use at temperatures in the range from 630.5° to 1,063° C. It is expected to provide temperature measurements of greater precision than is possible with the thermocouples now in use.

Experiments have been performed which show that it is possible to construct platinum resistance thermometers which have drifts of less than 0.001 deg/hr at 1,000° C. To achieve this stability, it was found necessary to use platinum wire of the highest available purity for the temperature-sensing resistor, to support the resistor in a strain-free manner, and to use materials containing no silica for support and protection.

In one successful thermometer design, four equally spaced synthetic sapphire disks provide support for the resistor wire. These disks are about $\frac{1}{2}$ cm in diameter. Eight relatively heavy lengths of platinum wire, 4 cm long, are threaded through holes in the disks. The wires are welded together at the ends to form a single continuous electrical conductor; its resistance at 0° C is 0.25 ohm. The complete resistor looks very much like a miniature elongated bird cage. Four leads of platinum wire, also supported by synthetic sapphire parts, are joined to the resistor, and the entire assembly is encased in a protecting tube of high-purity fused alumina, forming a thermometer about 45 cm long.

Subjecting the thermometer to mechanical shock tests showed that this design provides ample support for the platinum resistor. To test high temperature stability, the thermometer was heated for 100 hr at 1,000° C. During this period, the average temperature coefficient of resistance changed uniformly by an amount equivalent to a total drift of less than 0.1 deg at 1,000° C. A similar thermometer encased in a porcelain protecting tube exhibited a drift some 20 times as great.

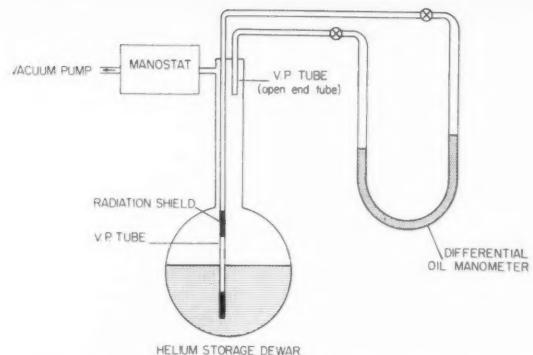
The problem of determining accurately the resistance of the thermometers is also being investigated. As part of this study, an improved Mueller resistance bridge was designed. Under evaluation is a potentiometric method of resistance determination which is independent of the lead resistance changes caused by varying temperature gradients along the thermometer. The electrical shunting effect of the synthetic sapphire parts introduces an error affecting the accuracy of resistance determinations. By carefully removing water and carbonaceous material from the thermometer, a total shunting resistance greater than 1 meg can be maintained at 1,100° C.

Precision Thermometry for Low Temperatures

THE BUREAU'S low-temperature program is being expanded in an attempt to provide high-precision thermometry in the range from 90° down to 2° K (-183° to -253° C), and to provide a calibration service for secondary thermometers from 20° down to 2° K. The measurement of low temperatures is growing in importance because of recent advances in cryogenic techniques, and because physicists and chemists need a practical and reliable working scale when they determine specific heats, thermal conductivities, and other fundamental properties of materials at these temperatures.

Although there is no international agreement on a practical temperature scale below 90° K, a gas thermometer may be used to make measurements on the thermodynamic temperature scale in this region. The inherent difficulties of this thermometer, however, make it impractical for use in regular calibration service. When stable platinum resistance thermometers were developed to operate in this range some years ago, the Bureau was able to establish a provisional scale from 90° down to 11° K. These thermometers are so stable that after 15 years of use, they have been found unchanged within the accuracy of their original calibrations. The provisional scale is based on a group of six of these thermometers, each of which was calibrated with reference to a helium gas thermometer.

Apparatus used in low-temperature research aimed at establishing an absolute temperature scale from 20° to 2° K (-253° to -271° C). George Cataland reads the differential oil manometer that indicates small differences in pressure within the liquid helium in the storage Dewar (lower right). Preliminary research has shown that helium vapor pressure must be controlled accurately if reproducible temperatures are to be achieved in this range.



Equipment used to measure and control the helium vapor pressure in a nearly constant-temperature liquid helium bath. Small changes in helium vapor pressure are measured by means of the differential oil manometer. A manostat is used to control the pressure in the helium storage Dewar.

The use of several thermometers to maintain the scale serves to show whether one of the standards has deteriorated. In the present calibration service from 11° to 90° K, resistance thermometers are compared (at 16 or more temperatures) with two of the Bureau's standards. This makes available a temperature scale which is believed to agree with the thermodynamic scale to an accuracy of 0.02° K.

In preliminary research toward making absolute temperature determinations from 20° down to 2° K, the Bureau studied changes in the velocity of sound in helium gas as a function of temperature in the region of 4.2° K. An acoustical interferometer was used for the first velocity measurements, and temperatures derived from the velocity of sound were compared with the temperature associated with the vapor pressure of the liquid helium bath surrounding the instrument. Although the results were encouraging, there was a need to know more about measuring helium vapor pressure and its association with liquid helium bath temperatures.

Recent Bureau investigations show that a nearly constant-temperature liquid helium bath can be achieved and associated with an extremely reproducible vapor pressure. The bath consists of a few liters of liquid helium in an ordinary metallic liquid-helium storage Dewar of 15- or 20-liters capacity. The vapor pressure of the liquid is controlled very accurately and liquid helium evaporates at a rate of approximately 300 cm³ per day. This technique will be of considerable help in determining the accuracy of temperatures derived from sound velocity measurements, because a better comparison will be possible between temperatures derived from vapor pressure and those derived from sound velocity.

When used in the constant-temperature liquid helium bath, carbon resistors displayed remarkable reproducibility of electrical resistance (resistances equivalent to temperatures ranging from a millidegree to a few tenths of a millidegree) versus helium vapor pressure in the range of from 2° to 4° K, even when the resistors had been cycled between 300° and 4° K. Several sources indicate that germanium resistors also exhibit excellent reproducibilities. Thus, both carbon and germanium resistors appear to be promising sources of secondary thermometers for use at low temperatures.

High-Temperature Gas Thermometry

THE Bureau has been studying electric arc techniques as sources of stable, controlled gas temperatures up to 20,000° K. Also under investigation are spectroscopic methods to measure reliably such high temperatures. These techniques are being studied as part of a program recently initiated to find simple, reliable methods of producing and measuring extreme temperatures and eventually to establish high temperature standards.

Temperature is a very useful parameter in analyzing many experimental physical and chemical processes. Increasing interest in very hot gases has made imperative a critical investigation of the methods currently available for producing and measuring temperatures in such gases.

The electric arc is a practical laboratory source of temperatures up to 20,000° K. Although the detailed mechanisms for current and heat conduction in a steady electric arc at atmospheric pressure and above are quite complicated, the current-carrying gas appears to be in approximate local thermodynamic equilibrium everywhere except in the vicinity of the electrode surfaces. Therefore, the gas temperatures, in the usual sense of the word, can be calculated from the gas compositions determined from spectroscopic measurements. Transport properties and chemical equilibria can also be obtained for gaseous mixtures at these high temperatures from complete composition data over the cross section of an electric arc.

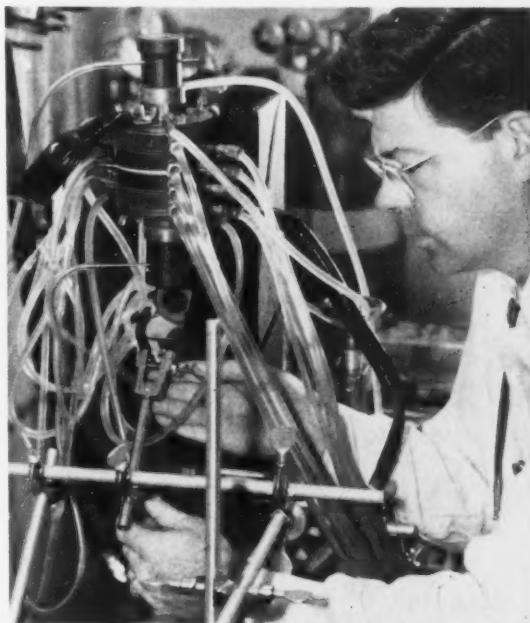
A device for maintaining a controlled electric arc in an atmosphere of any desired composition has been built. It consists of a stack of water-cooled copper washers alternating with electrically insulating bakelite washers. The arc burns in the central cylindrical core of this stack of washers between electrodes at each end. The gas to be studied is introduced into the core of the arc. Windows are provided along the sides and at the ends of the apparatus for optical viewing and spectrographic analysis of the radiation from the arc. Research at the present time with this equipment is concerned mainly with comparisons between photoelectric and photographic measurement methods; comparisons between observation from the side and along the arc axis; and with effects of optical geometry, arc length, current, and similar easily varied parameters in an effort to determine their importance to the precision and accuracy of temperature measurements.

Information and techniques that are gained from helium vapor pressure investigations will be applied toward determining vapor pressures of other liquids at higher temperatures, thus improving current knowledge of their relationships with temperatures. Also included in the low-temperature program will be studies of transitions in pure substances. It is expected that these studies will make possible a choice of accurately determined transition temperatures to serve as fixed points for calibrating thermometers, and to provide a basis for agreement on an international temperature scale below 90° K.

High-Temperature Gas Thermometry

An important experimental difficulty is that of obtaining suitable high-intensity light standards for use in measuring absolute intensities of the arc spectroscopic lines. Therefore, concurrently with the research on arcs, high temperature continuum radiation standards are being improved. A carbon tube heated directly by a heavy current has been developed and seems promising as a constant radiation standard up to a temperature of about 3,000° K. Different construction materials may permit even higher temperatures. Currently under construction is a very-high-pressure free-burning arc which for some purposes may provide a suitable standard of continuum radiation corresponding to temperatures of 10,000° K or more.

Charles R. Yokley views the high-temperature furnace developed at the Bureau for maintaining a controlled electric arc in an atmosphere of any desired composition. The arc burns in the central cylindrical cavity of the stack of washers. Windows are provided for viewing and spectrographic analysis of the radiation from the arc.



THE Bureau is now distributing 60,000 samples of standard materials a year to other laboratories for use in controlling chemical processes and in maintaining the accuracy of apparatus and equipment. A total of over 600 different standard materials are available from the Bureau—principally chemicals, ceramics, metals, ores, and radioactive nuclides.¹ All are certified either for chemical composition or with respect to a specific physical or chemical property such as melting point, viscosity, or index of refraction. These standards make possible uniform measurements of heat and temperature, define the colors of paints, and calibrate instruments that control the composition of metals and motor fuels.

Within the past 5 years, requests from American science and industry have resulted in the issuance of about 70 new standard materials. Meanwhile, 150 new standard materials are being prepared for issuance through programs of careful analysis and precise measurement. Much of this expansion in the standard samples program has resulted from current efforts by Government and industry to develop materials having specific properties for use at high temperatures or under other extreme conditions.

Chemical Standard Samples

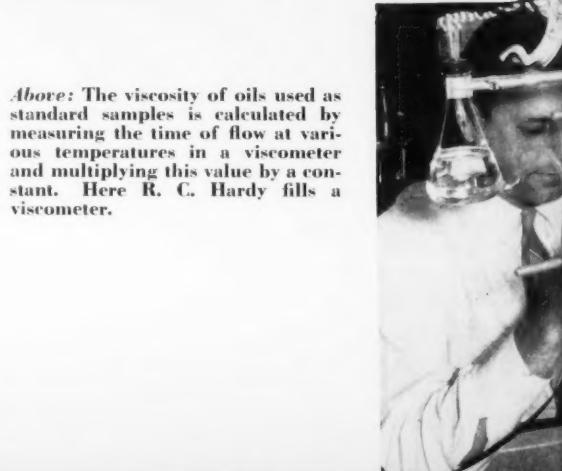
The standard samples program was established in 1905 when the American Foundrymen's Association turned over to the Bureau four sets of cast iron to be standardized for chemical composition. The majority of these chemical standards are metals and alloys, used extensively for monitoring the thousands of analyses made daily in industrial laboratories. Standards of chemical composition also serve as guides in developing new analytical methods or determining the composition of unknown materials. New chemical standards are prepared whenever an industrial need develops. Recent additions include high-temperature cobalt-nickel alloys for jet and missile production, titanium alloys for aircraft and ordnance research, zirconium alloys for nuclear-power development, lithium ores for the new lithium chemical industry, lead-tin bronzes for Navy defense purposes, and portland cement for the cement manufacturers.

About 200 samples of hydrocarbons are issued as "pure substances" and certified with respect to their degree of purity (99.99%). This category was initiated to fill the gap for standards needed to calibrate instruments, particularly mass spectrometers, employed in controlling the production of synthetic rubber and of special fuels for military aviation. These samples may also find application as gas chromatographic standards. As one of this group, a halogenated hydrocarbon—bromobenzene—is being purified. This compound will be particularly useful in the rubber industry.

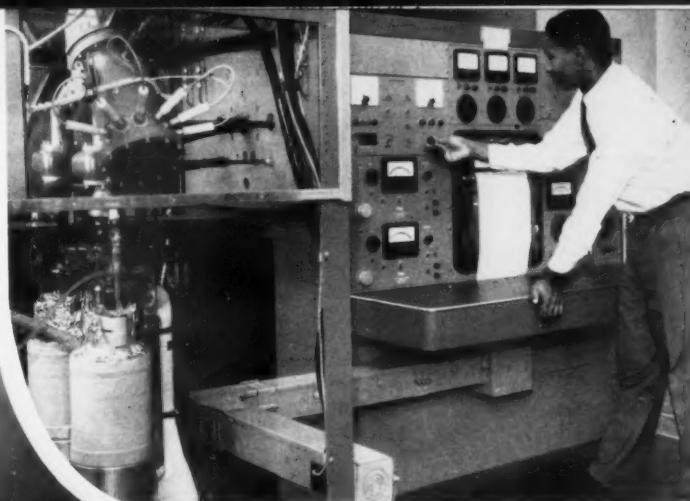
In connection with the hydrocarbon program, a new group of standard samples is being developed. Each of these standards will be a mixture of pure hydrocarbons—rather than one pure compound—and will be used mainly for checking gasoline and petroleum fractions. At present, eight such mixtures are under consideration.

STANDARD MATERIALS EXPANDED

Right: Richard Flitsch takes data from an X-ray fluorescence spectrometer used to analyze standards for uniformity. A major use of the standard is for maintaining accuracy of equipment of this type.



Above: The viscosity of oils used as standard samples is calculated by measuring the time of flow at various temperatures in a viscometer and multiplying this value by a constant. Here R. C. Hardy fills a viscometer.



Above: Minor isotopic abundances of uranium standards are determined by surface-ionization mass spectrometers. E. L. Garner checks the tube pressure during an analysis.



Left: J. L. Shultz analyzes a standard metal sample for chromium, vanadium, and manganese by potentiometric titration. *Above:* To minimize X-radiation, standards of radiation are stored in a specially built chamber. L. L. Stockmann measures the level of radiation with a Geiger counter.

A new set of metal standard samples comprises seven types of steels whose oxygen and nitrogen content have been carefully determined. These reference materials are necessary for the calibration of analytical equipment used to determine the gaseous elements in various kinds of commercial steels. Low gas content is a characteristic of metals with desirable properties, and the acceptable amount of gases present in a metal may be expected to be stipulated in procurement specifications for metals and metal products in the future. With the certification of these standards, the Bureau has begun a program for producing gases-in-metals standards for a number of metallurgical products. Work is now in process to develop standard samples certified for the content of oxygen and hydrogen in titanium alloys.

Spectroscopic Standard Samples

High-speed spectrochemical methods of analysis have been extensively adopted in recent years by both producers and consumers of metal products. These methods are essentially comparison techniques in which the spectra from unknown samples are referred to the spectra from samples of known composition. Thus standard samples play a basic role in spectrochemical analysis.

The first spectroscopic samples were developed during the last war in response to a need for standards for high-speed control analysis in steel production and inspection. To date, about 120 of the composition standards have been specifically produced for spectroscopic use. Metals and alloys for 44 new spectroscopic standards have been prepared and are being analyzed for content and tested for homogeneity by Bureau scientists, while final plans have been made for 66 others yet to be prepared.

About 3 years ago, after studying industrial and government needs, the Bureau started a greatly expanded program on spectroscopic samples. This pro-

Tabulation of NBS Standard Materials

Composition standards			Other standards	
Chemical:				
Steels	46	Spectrographic:	95	Hydrocarbons
Irons	9	Steels	1	Radioactive
Ferroalloys	8	Zn alloys	1	Paint pigments
Nonferrous	19	Tins	10	Oils, viscosity
Ores	12	Nickel oxide	3	pH chemicals
Ceramics	19	High-temperature	4	Melting point materials
Primary chemicals	14	Ti alloys	3	Rubber materials
Uranium isotopes	15	Total	123	Phosphors
Total	142			Other ref materials
				Total
			616	351
		Total		

gram is designed not only to satisfy basic requirements of industry and Government for such common materials as cast iron, steel, and copper-base alloys, but also to provide standards of newer materials—such as high-temperature alloys, titanium alloys, and zirconium alloys—the former two finding application in the aircraft and missile industrial fields and the latter in atomic energy installations. The Department of Defense, Atomic Energy Commission (AEC), and industrial groups have assisted in selecting the types of alloys needed for these standards and in establishing priorities for their application.

Four corrosion-resistant, high-temperature standards: 19-9DL, AMS 5360A (AISI 316), AMS 5376A (N-155), and Nimonic 30a; and 3 titanium standards are now available, while a total of 18 other high-temperature standards and 6 zirconium-base standards will be certified in the near future.

To improve specific physical properties of metallurgical alloys, minute amounts of certain elements are added. Thus the heat-resistant alloys used in rockets and jet engines contain various combinations of boron, tungsten, molybdenum, titanium, zirconium, niobium, tantalum, and aluminum as well as the major metals. Because of the complex composition of these materials and difficulties in chemical analyses, spectrographic and X-ray methods are used to control their quality in production. The Bureau has been preparing standards which are certified for trace elements as well as major constituents. For instance, in a set of eight ingot iron and low-alloy steels recently prepared, 17 elements are included in provisional cer-

Highly purified benzoic acid crystallized in thermometric cells provides a precise standard for calibration of resistance thermometers. Here D. Enagonio prepares to cool a hot supersaturated solution just removed from the oven.



tificates of analysis, and six more are under study for further certification. Another example is the new set of zirconium standards which is being developed primarily for application in the atomic-energy field. Some 30 elements are under investigation in this set—many at the parts-per-million (ppm) level.

The field of X-ray fluorescence has become increasingly important for rapid analysis. Like optical-emission instrumentation, these newer X-ray instruments require standard samples for their calibration. The Bureau recognized this need several years ago and many spectroscopic standards which are now avail-

POINT-SOURCE GAMMA-RAY STANDARDS are now available for the first time. These standards are gamma-ray emitters and will be useful to research workers in approximating "ideal" conditions when calibrating scintillation counters and other beam-detecting devices.

Five of these standards are being issued under the Bureau's program to supply standards in all areas of the physical sciences. The five are sodium-22, zinc-65, niobium-95, strontium-85, and mercury-203. Each standard consists of radioactive material deposited between two layers of a thin polyester tape.

The activity of each of the nuclides distributed in this form is approximately 5×10^4 gamma rays per second. Sodium-22, which has a half-life of 2.6 years, emits a gamma-ray of 1.28 Mev energy, an annihilation gamma ray of 0.511 Mev energy, and X-radiation characteristic of neon. Zinc-65, with a 45-day half-life, gives off a 1.11-Mev gamma ray, an annihilation gamma ray of 0.511 Mev, and X-rays characteristic of copper. Strontium-85 emits a gamma ray with an energy of 0.513 Mev and X-radiation characteristic of rubidium. Its half-life is 65 days. Niobium-95 which has a half-life of 35 days, emits a 0.768-Mev gamma ray. Mercury-203 emits a 0.279-Mev gamma ray and X-radiation characteristic of thallium. Mercury-203 has a half-life of 46.5 days.

The standards cost \$27.00 each and are available from the Radioactivity Section, National Bureau of Standards, Washington 25, D.C.

able or in preparation are designed for X-ray fluorescence analysis. To date 14 standard samples have been developed specifically for X-ray fluorescence while 12 standards are equally suitable for both methods of analysis.

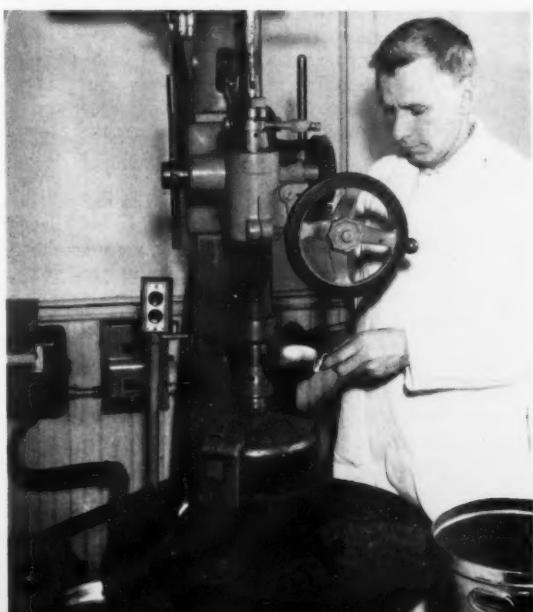
Radioactive Standard Samples

Since 1913, when the United States received its first national radium standard (Radium-6), much of the radium produced or sold in this country has been calibrated against this standard by the Bureau. With the production of radioactive materials in the 1930's, the need for radioactivity standards of elements other than radium has steadily increased, and in fact, has been accelerated by the discovery of controlled nuclear fission. For radiation safety, for planning experiments, and for ordering and disposing of active material, a knowledge of the disintegration rate of the specimen is essential. Hence, the total efficiencies of the various radiation detectors under differing experimental conditions must be accurately determined by calibration against radioactive samples.

Today the Bureau distributes standard samples of 40 different types of radioactive nuclides. These standards are used for the calibration of equipment which in turn are used in the measurement of the same types of nuclides. These nuclides have widely varying applications. Several are used medically in the treatment of such ailments as leukemia (phosphorus-32) and cancer (gold-98 and others), as well as in the study of blood formation (iodine-131) and metabolism (carbon-14). Cobalt-60 is used in munitions plants to check flaws in casting parts or shell casings. Other nuclides are used to determine such factors as contamination of food crops by strontium-90 and the age of wine and liquors as indicated by the amount of tritiated water (water containing hydrogen-3) present.

Standard samples of mercury-203, niobium-95, and

Cuttings for an NBS metal sample are broken up into a fine size by crushing in a chilled-iron grinder, operated by G. E. Deardorff.



R. K. Bell analyzes a standard metal sample for antimony, arsenic, and tin in a distillation apparatus.

strontium-85 have just been made available for distribution, while 5 point-source standards—including sodium-22 and zinc-65—are being prepared.

Uranium Standards

The considerable expansion in the application of uranium and related atomic-energy materials in recent years has emphasized the need of uniform standards which will be widely accepted for standardizing materials and calibrating instruments. Although various AEC laboratories and contractors have developed standard materials for their own use, a central set of standards was needed.

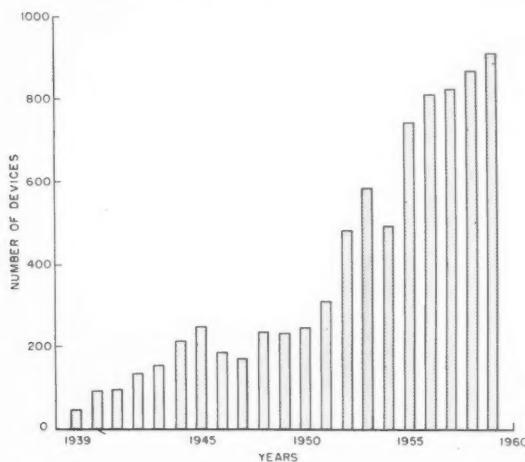
To meet this demand, the Bureau issues a series of 15 isotopic standards in cooperation with AEC. This set, used by educational and research institutions and industries both in the United States and abroad, contains a graduated series of standard samples ranging from uranium depleted in U^{235} (containing 0.5% U^{235}) to a highly enriched material containing 93 percent U^{235} . Selection of the composition of the intermediate samples was made upon consultation with an advisory committee set up by AEC. These standards of uranium enable laboratories to evaluate their own reference materials.

In addition to the isotopic range, a natural uranium standard is included in the series. This standard is often used directly as a working standard or control material in experiments. The program is being expanded to include standards for other atomic-energy materials such as plutonium and thorium.

A complete listing of standard materials issued by the National Bureau of Standards is contained in NBS Circular 552 (3rd edition), Standard Materials, which is available from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C. (35 cents).

Million-Pound Dead-Weight Machine Designed

FINAL designs are nearly completed for dead-weight machines of 300,000 and 1,000,000-lb capacity which will be installed at the new location (Gaithersburg, Maryland) of the National Bureau of Standards. These machines will be used to calibrate force-measuring devices that are used, in turn, to calibrate other devices and machines for measuring and applying large forces. Force measurements are widely used in industrial fields such as weighing, materials testing, automatic control of machinery, and rocket development, and there is a consequent need for precisely measuring forces larger than ever before.



Calibrations of force-measuring devices submitted to the Bureau from 1939 to 1959.

By use of dead weights totaling 111,000 lb, the Bureau now calibrates force-measuring devices to an accuracy of 0.02 percent. The Bureau's largest dead-weight machine had a capacity of 102,000 lb when installed in 1927. Nine 1,000-lb weights were added to this machine in 1931, thereby increasing its capacity to its present limit of 111,000 lb.

When this machine was installed, there were few devices capable of measuring 100,000 lb. Since that time, there has been an increasing number of larger force-measuring devices, some as large as 3,000,000-lb capacity. From only a few in 1927, the number of force-measuring devices submitted to the Bureau for calibration has grown to more than 900 in 1959. Devices measuring forces over 111,000 lb are calibrated by the Bureau by means of equipment that have been calibrated previously in the dead-weight machine.

To calibrate devices that measure forces greater than the capacity of the dead-weight machine, several steps are required, and errors are introduced, thereby lower-

ing calibration accuracy. Load cells of 3,000,000-lb capacity are now calibrated to an accuracy of 0.4 percent in the Bureau's 10,000,000-lb compression machine. This accuracy, however, is not sufficient for the needs of modern industry.

Industrial weighings require accuracies of 0.05 to 0.2 percent; and accuracies of 0.1 percent or better are now needed for measuring thrusts of large rocket engines. Calibration accuracies with the new machines should be well within these requirements. Weights for the machines are expected to be accurate within 0.005 percent. Temperature, too, will be controlled carefully. Because the indication of a force-measuring device depends on the temperature, provisions will be made for determining their temperature coefficients by calibrating them at temperatures from 50° to 100°F.

Although the Bureau now calibrates tension-measuring devices only to 111,000 lb, it will be able to calibrate devices in both tension and compression to 1,000,000 lb in the new machines. These additional capabilities should greatly accelerate the extension of modern force-measuring methods.

Vacuum Standards Program Enlarged

AS PART of its program in pressure measurements, the Bureau has established a Vacuum Standards Laboratory in which it will concentrate its work on developing absolute standards for pressures in the vacuum range and investigating useful secondary standards. Also included in the program will be a study whose aim will be the generation of more reproducible pressures in this range to serve as fixed points, and to effect necessary improvement in accuracy of measurements by different organizations and scientific workers.

High-vacuum pressures correspond to the pressures encountered by missiles and space vehicles in flights to altitudes of many miles above the earth's surface, and there is a growing need for precise measurement in this pressure range. Improved measurement of extremely low pressures also is needed in investigating adsorption phenomena and other surface properties of materials, as well as in operating equipment such as particle accelerators used in studies of nuclear reactions.

High-Pressure Standards Program Expanded

THE Bureau has expanded its pressure standards program to include development of improved standards and techniques for measuring very high pressures. Changes in the properties of materials will be studied to provide more precisely determined calibration points on the pressure scale, and techniques for measuring high pressure will be investigated.

High pressures offer great promise in treating new materials to meet some of the most severe military and industrial requirements. For example, high pressures cause chemical changes that form new compounds such as polyethylene and boron nitride, and crystalline changes such as occur when graphite is changed to diamond, or quartz to Coesite.

Remarkable changes in physical properties often appear when materials are subjected to high pressures.¹ Some familiar electrical insulators become semiconductors, and some familiar semiconductors become conductors. Brittle substances such as bismuth and quartz become ductile. Tungsten carbide more than doubles in strength when subjected to a hydrostatic pressure of 400,000 psi.

Above 500,000 psi, experimental techniques differ from those at lower pressures because nearly all fluid materials become solid. For example, at room temperature water solidifies at 140,000 psi, mercury at 180,000 psi, and nitrogen at 400,000 psi. In studying materials at very high pressures, the pressure-transmitting media now used are solids that have low shear strength, such as indium, lead, tin, silver chloride, talc, or pyrophyllite.

In the high-pressure program, effort will be directed toward the extension to higher pressures of those techniques that are now used to generate and measure pressures below 200,000 psi. These pressures can now be measured by dead-weight-loaded pistons that are not packed to prevent pressure fluid from leaking. Instead, the clearance between piston and cylinder is made so small that leakage is kept within a few cubic inches per month. This type of apparatus may be found usable, with a fluid pressure medium, at pressures as high as 350,000 or 400,000 psi.

The program will include attempts to furnish more precisely determined basic information. For measurements of pressures above 200,000 psi, scientists now refer to approximately known pressures at which there are changes in phase, volume, or electrical resistance of certain materials. The resistance of bismuth, for example, changes abruptly at 365,000, 385,000, and 1,700,000 psi; of thallium at 630,000, and of barium at 1,100,000 psi. Because these values were obtained with different types of apparatus at widely different times, there may be inconsistencies between them.

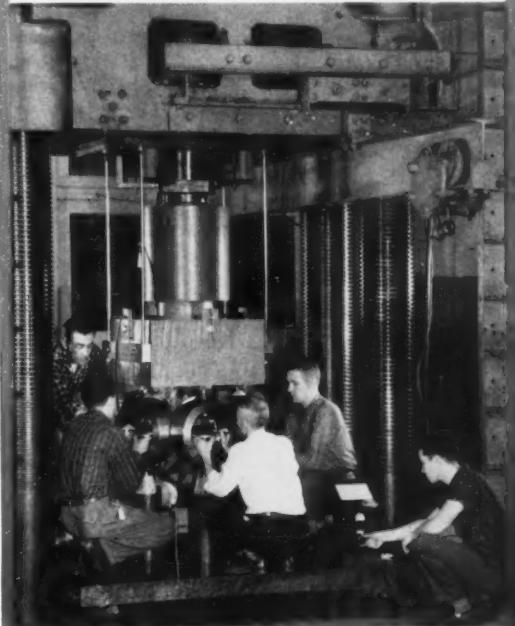
Various transitions will be studied by use of the Bureau's tetrahedral anvil apparatus,² (see article next

page) as well as with high-pressure piston-and-cylinder equipment, in an attempt to adjust possible inconsistencies. By refining measurement techniques, Bureau scientists will attempt to improve the accuracy of pressure values at transition points so that they can be used in pressure calibrations.

¹ High-pressure effects on solids studied by infrared spectroscopy, *NBS Tech. News Bul.* **43**, 138 (July 1959).

² Compact multi-anvil wedge-type high pressure apparatus, by E. C. Lloyd, U. O. Hutton, and D. P. Johnson, *J. Research NBS* **63C**, 59 (1959).

Calibration of 1,500,000-lb-capacity load cell (vertical cylinder above large block). This load cell will be part of the instrumentation used in tests of a 1,500,000-lb-thrust rocket engine that is being developed for use in space exploration. Each of the five 300,000-lb-capacity proving rings (below large block) has been calibrated by dead weights to 110,000 lb, and by other calibrated proving rings for loads greater than 110,000 lb.



Compact Apparatus for Generating High Pressures

AN EXTREMELY compact apparatus¹ for generating high pressures has been designed by E. C. Lloyd, U. O. Hutton, and D. P. Johnson. The apparatus produces pressures of 100,000 atm and above by the application of force against each face of a $1\frac{1}{2}$ -in. regular tetrahedron of pyrophyllite (hydrous aluminum silicate). The device was made to enable Bureau scientists to study the properties of materials at high pressure so that "fixed points" can be established on the pressure scale, and so that improved pressure measurement techniques can be devised.

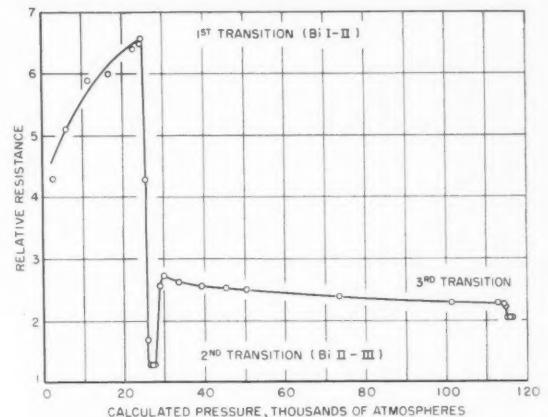
Other devices for a similar purpose have been made² with 4 independent hydraulic rams to apply forces to the faces of a tetrahedron. The Bureau design, however, consists of an assembly in which external force is applied to only 1 of 4 anvils, with wedge reaction forces acting on the remaining 3 anvils. Because only one external force is needed to operate it, the apparatus can be used in a conventional hydraulic press. The apparatus has a diameter of only 8 in. at its lower bolster.

While a specimen is under high pressure, four electrical connections through the anvils permit resistance heating of the specimen and measurement of its temperature, electrical resistance, or other quantities. Other advantages of the apparatus are easy manipulation and alinement of the anvils, and rapid assembly and disassembly.

Construction and Operation

The instrument consists essentially of 4 anvils having tungsten carbide tips that bear on the faces of a tetrahedron of pressure-transmitting material (pyrophyllite), in which a specimen is embedded. The tip of each anvil is ground to a flat triangular face and transmits pressure to one of the faces of the tetrahedron. The vertical anvil is forced downward by the press, and the remaining anvils transmit reaction forces from a conical retaining ring on which the three lower anvils are seated. Thus, forces are applied to the tetrahedron from four directions. These forces are very nearly

Wedge-type high-pressure apparatus in hydraulic press. This particular model was recently completed to investigate the advantages of larger size. U. O. Hutton positions upper bolster plate on apparatus designed to apply force to each side of 1-in. tetrahedrons.



Changes in electrical resistance of bismuth determined by use of tetrahedral pressure cells. Properties of materials of high pressure are under study, with the object of establishing "fixed points" on the pressure scale.

equal, and there is substantially equal motion of each anvil toward the center of the tetrahedron.

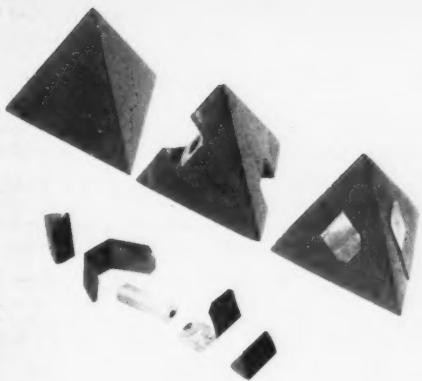
The retaining ring and the anvils (excluding the tips) are made of SAE 4340 steel heat-treated to Rockwell 40/41C. Repeated applications of 100-ton loads have resulted in no permanent deformation to either the anvils or the retaining ring.

The inner surface of the retaining ring makes an angle with the horizontal that is nearly the same as the angle between the faces of the tetrahedron (70.528 deg). This surface is inclined approximately 1 deg more, however, to compensate for the friction (coefficient of friction approximately 0.01 with Teflon lubricant) between the butt ends of the anvils and the conical surface. This friction is held to a minimum for satisfactory operation.

Several materials have a low coefficient of friction under heavy loading and appear suitable for use as lubricants between these surfaces. Sheet Teflon, 0.003-in. thick, was found to be satisfactory and it also provides the electrical insulation that is needed between the anvils and the ring when studies are made of changes in electrical resistance with pressure.

Electrical Resistance Studies

When the apparatus is used to study resistance changes with pressure, a specimen is first inserted in a hole (up to $1\frac{1}{8}$ -in. diam.) drilled from edge to edge of the tetrahedron. Pieces of silver foil are placed in contact with each end of the specimen, and bent to give an exposed surface to each of the anvil faces. Thus, one end of each foil is in position to make electrical contact with one anvil. The remaining notches in the edges of the tetrahedron are then filled with prisms of pyrophyllite, and the tetrahedral assembly



Pyrophyllite tetrahedrons and details of pressure cell assembly used in apparatus generating pressures of 100,000 atm and above. Tetrahedron shows notches and hole cut to receive specimen. Specimen (in sheath of silver chloride), silver foils, and prisms to fill notches are shown in the relative positions they occupy when assembled in pressure cell.

is ready for an experiment. This arrangement makes available an electrical lead via each of the 4 anvils. Thus, a 4-lead circuit can be used for resistance measurements during studies of changes in electrical resistance with increasing pressure.

Measurements of relative resistance with pressure have been made with specimens of antimony, barium, and bismuth. The results have shown good agreement with the work of other investigators. Values of ram forces required to reach the first resistance transitions of barium and bismuth have indicated that approximately 10 percent of the total applied force is lost to gasket forces and to the internal friction of pyrophyllite.

Future Investigations

It appears desirable to investigate other pressure-transmitting materials for use as tetrahedral pressure cells. Also, certain combinations of tetrahedron material and specimen sheath material may have more nearly optimum properties and may improve the uniformity of pressure applied to a specimen.

If a larger tetrahedron were used for a given specimen size, more uniform pressure would result, and the proportion of force taken by the gasket should be re-



Larger model of multi-anvil apparatus built for applying loads up to 600 tons to tetrahedrons of about 1-in. edge length.

duced. Recently, a larger apparatus of the same general design has been built so that the advantages of larger size may be investigated. The new apparatus is designed to apply forces up to 600 tons to tetrahedrons of about 1-in. edge length. Success in operating the larger apparatus would indicate the feasibility of a 2-stage device, in which a small tetrahedron would be embedded in a tetrahedron of, say, 3 in. on a side. The forces needed to support the second-stage anvil would be supplied by the pressure in the large tetrahedron.

It is hoped that further work will enable the Bureau to determine more accurately the pressures at which there are changes in electrical resistance, changes of state, and polymorphic crystalline transitions in certain materials. The properties of these materials could then be used to define fixed pressure points useful in pressure calibrations, in much the same way that freezing points of certain materials are used to define the International Temperature Scale.

¹For further information, see Compact multi-anvil wedge-type high pressure apparatus, by E. C. Lloyd, U. O. Hutton, and D. P. Johnson, *J. Research NBS* **63C** 59 (1959).

²Some high-pressure, high-temperature apparatus design considerations: equipment for use at 100,000 atmospheres and 3,000° C., H. Tracy Hall, *Rev. Sci. Instr.* **29**, 267 (1958).

A Bibliography of NBS Publications of Interest to Other Standards Laboratories

THREE means by which the results of Bureau research are disseminated to the scientific and technological community. One means is by direct contact between the Bureau's technical staff and scientists, measurements specialists, and quality control personnel in other laboratories. A second means is by providing technical assistance and advice to secondary

standards laboratories in industry, government, and testing institutions. A third means is by direct dissemination of measurement standards through NBS calibration of master instruments used in technological institutions and through the sale of reference materials to industrial organizations and scientific laboratories. A principal means is that of publication in professional

journals and in the Bureau's own regular periodicals, monographs, and other series.

When not otherwise indicated, these publications may be purchased from the Superintendent of Documents, Government Printing Office, Washington 25, D.C. Those indicated as available from the Bureau are distributed by the Publications Section, Office of Technical Information, National Bureau of Standards, Washington 25, D.C. Bureau publications are also available in the principal university and public libraries that serve as Depository Libraries for Government documents.

General

Assistance to other standards laboratories. NBS Tech. News Bull. **43**, 21 (February 1959).

Publications of the National Bureau of Standards 1901 to June 30, 1947, with subject and author indexes. NBS Circ. **460** (1948), 375 pp, price \$1.25.

Publications of the National Bureau of Standards July 1, 1947 to June 30, 1957, with subject and author indexes. Supplement to NBS Circ. **460** (1958), 373 pp, price \$1.50. (Purchasers of this publication who fill out and submit the blank form that accompanies it are sent semiannual supplementary lists of NBS publications at no additional cost.)

Organization and functions of the National Bureau of Standards. Department of Commerce Order No. 90 (revised), reprinted from *Federal Register* (June 11, 1958). Available from NBS Publications Section.

Test fee schedules of the National Bureau of Standards, published when issued in *Federal Register*. Reprints available from NBS Publications Section.

Research highlights of the National Bureau of Standards (Annual Report, 1958), Miscellaneous Pub. 226, price 45¢ for 1957, No. 223, price 45¢.

Standard materials issued by the National Bureau of Standards, NBS Circ. **552** (3d ed., 1959), 27 pp. Price 35¢, from Superintendent of Documents. Supplementary insert sheets, issued from time to time, are available without charge from the NBS Publications Section.

Mass; Length; Volume

Units and systems of weights and measures: their origin, development, and present status. by L. V. Judson. NBS Circ. **570** (1956) 29 pp, price 25¢.

Precision laboratory standards of mass and laboratory weights, by T. W. Lashoff and L. B. Macurdy. NBS Circ. **547**, section 1 (1954) 24 pp, price 25¢.

Calibration of line standards of length and measuring tapes at the National Bureau of Standards, by L. V. Judson. NBS Circ. **572** (1956) 11 pp, price 25¢.

The measurement of thickness, by G. Keinath. NBS Circ. **585** (1958) 79 pp, price 50¢.

Testing of glass volumetric apparatus, by J. C. Hughes. NBS Circ. **602** (1959) 14 pp, price 20¢.

Pressure; Density; Humidity; Gas Flow

Testing of precision manometers and barometers, by W. G. Brombacher, J. L. Cross, and D. P. Johnson. NBS Mono. **8** (in press).

Density of solids and liquids, by P. Hidnert and E. L. Peffer. NBS Circ. **487** (1950) 29 pp, price 20¢.

Testing of hydrometers, by J. C. Hughes. NBS Circ. **555** (1954) 10 pp, price 10¢.

Electric hygrometers, by A. Wexler. NBS Circ. **586** (1957) 21 pp, price 20¢.

Methods of measuring humidity and testing hygrometers, by A. Wexler and W. G. Brombacher. NBS Circ. **512** (1951) 18 pp, price 15¢.

Determination and correlation of flow capacities of pneumatic components, by D. H. Tsai and M. M. Slawsky. NBS Circ. **588** (1957) 7 pp, 10¢.

Heat; Temperature; Thermal Expansion

Instruction manual for precise measurement of heat of combustion with a bomb calorimeter, by R. S. Jessup. NBS Mono. **7** (in press).

Calibration of liquid-in-glass thermometers, by J. F. Swindells. NBS Circ. **600** (1959) 21 pp, price 20¢.

Methods of testing thermocouples and thermocouple materials, by W. F. Roesser and S. T. Lonberger. NBS Circ. **590** (1958) 21 pp, price 20¢.

Reference tables for thermocouples, by H. Shenker, J. I. Lauritsen, Jr., R. J. Corruccini, and S. T. Lonberger. NBS Circ. **561** (1955) 84 pp, price 55¢.

Thermal expansion of solids, by P. Hidnert and W. Souder. NBS Circ. **486** (1950) 29 pp, price 20¢.

Light; Radioactivity; X-rays

Spectrophotometry (200 to 1,000 millimicrons), by K. S. Gibson. NBS Circ. **484** (1949) 48 pp, price 35¢.

Colorimetry, by D. B. Judd. NBS Circ. **478** (1950) 56 pp, price 40¢.

Photoelectric tristimulus colorimetry with three filters, by R. S. Hunter. NBS Circ. **429** (1942) 46 pp, price 20¢.

Preparation, maintenance, and application of standards of radioactivity, by W. B. Mann and H. H. Seliger. NBS Circ. **594** (1958) 47 pp, price 35¢.

Design of free-air ionization chambers, by H. O. Wyckoff and F. H. Attix. NBS Handbook **64** (1957) 16 pp, price 20¢.

Electrical Measurements

Suggested practices for electrical standardizing laboratories, by F. B. Silsbee. NBS Circ. **578** (1956) 9 pp, price 15¢.

Establishment and maintenance of the electrical units, by F. B. Silsbee. NBS Circ. **475** (1949) 38 pp, price 25¢.

Extension and dissemination of the electrical and magnetic units by the National Bureau of Standards, by F. B. Silsbee. NBS Circ. **531** (1952) 33 pp, price 25¢.

Precision resistors and their measurement, by J. L. Thomas. NBS Circ. **470** (1948) 32 pp, price 20¢.

High-frequency voltage measurements, by M. C. Selby. NBS Circ. **481** (1949) 14 pp, price 20¢.

Radio-frequency power measurements, R. A. Schrack. NBS Circ. **536** (1953) 16 pp, price 20¢.

Techniques for accurate measurement of antenna gain, by H. V. Cottony. NBS Circ. **598** (1958) 10 pp, price 15¢.

Calibration of commercial radio field-strength meters at the National Bureau of Standards, by F. M. Greene. NBS Circ. **517** (1951) 5 pp, price 10¢.

Attention is also called to the following papers which, though not published by the Bureau, were written by NBS staff members and describe some recent developments. All appear in IRE Transactions on Instrumentation, Vol. **I-7**, Nos. 3 and 4 (double issue) December 1958, published by the Professional Group on Instrumentation (PGI), Institute of Radio Engineers, 1 E. 79th St., New York 21, N.Y.; price to nonmembers \$3.00, to IRE members \$1.50, to IRE-PIG members \$1.00:

The ammonia maser as an atomic frequency and time standard, by R. C. Mockler, J. Barnes, R. Beehler, H. Salazar, and L. Fey.

High-frequency standards of the NBS Electronic Calibration Center, by M. C. Selby.

High-frequency impedance standards at the National Bureau of Standards, by R. C. Powell, R. M. Jickling, and A. E. Hess.

A dry, static calorimeter for RF power measurement, by P. A. Hudson and C. M. Allred.

Recent developments in the field of microwave power measurements at the National Bureau of Standards, by G. F. Engen.

Recently developed microwave impedance standards and methods of measurement, by R. W. Beatty and D. M. Kerns.

Mechanizing Calibration Activities

IN an effort to cope with the increasing backlog of calibration work, the Bureau has been exploring specific possibilities for automating many of the data-recording and processing tasks encountered in a wide variety of calibration programs. By using high-speed computers for some of the routine data-handling aspects of calibration activities, savings of time and labor, as well as increased quantity and quality of work output, can be realized. An example is the automatic calibration of gage blocks. The large volume of test data resulting from gage-block comparisons is now being programmed for the Bureau's high-speed electronic computer. The computer automatically calculates and prints out the final corrections.

A number of other calibration areas are now being aided by automatic data-processing techniques or are being considered for such techniques. For instance, in the production of standard metallic samples, three spectrometers list the spectral line intensities of alloys either on punched cards or on perforated tape; these

data are subsequently fed into an electronic computer for printing out the compositions. In measurements of X-ray coefficients in pure materials, the 1½ man-years of data reduction formerly resulting from three man-weeks of experimentation has been materially shortened by systematic transfer of spectral data to perforated tape and thence to a computer. The testing of glass color samples is to be programmed to allow for the special characteristics of the light source and prism: transmissivity at specific wavelengths will be recorded digitally on perforated tape and converted to tristimulus values. The calibration of standard platinum resistance thermometers is to be mechanized, from read-out of the resistance measuring bridges to printing of the certificates. The paths of light rays refracted through an aerial camera lens, and intersecting a selected focal plane, are already being computed digitally and converted to an image pattern; it is expected that ultimately an objective judgment of the lens quality will be made by machine from this pattern.

Confirmation of the Rydberg Constant

THE Bureau has reevaluated data used in an early determination of the Rydberg constant, increasing accuracy by a factor of 10, and bringing this earlier calculation into line with the presently accepted value.¹ This work, by W. C. Martin of the Bureau staff, constitutes, in effect, an independent confirmation of the value of the Rydberg constant, which since 1952 has been based on the work of only one set of observers.

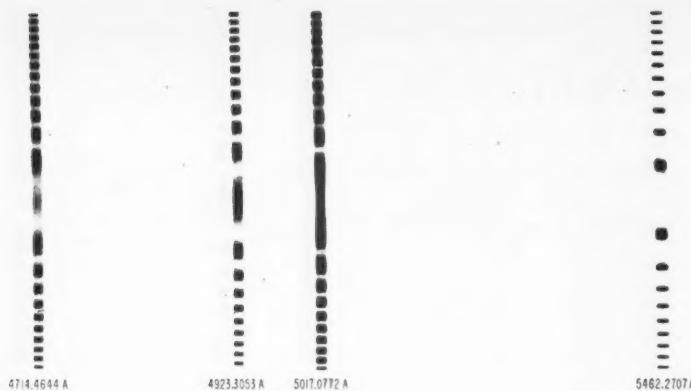
The Rydberg constant, $R\infty$, which relates the energy levels in atoms, enters into the frequency or wave-number formulas for all series in atomic spectra. It is at present the most accurately known combination of fundamental atomic constants: a simple formula links its value to the mass of the electron m , the electron charge e , the speed of light c , and Planck's constant h . The "primitive," or less accurately known constants, such as h , are adjusted to satisfy the value of $R\infty$. Because of the importance of the Rydberg constant, employing a value that had been directly confirmed by only one group was very unsatisfactory. The Bureau therefore undertook the present work, which has succeeded in putting the Rydberg constant on a firmer footing.

This evaluation was carried out as part of a broader program which seeks to obtain more accurate values for important physical constants, such as the velocity of light, the acceleration of gravity, and the various atomic constants. These constants of nature, when determined to extremely high accuracy, provide invari-

ant bases for the reproduction of standards of many physical quantities such as length, time, and electric current. They thus serve to lock present standards and units of physical measurement into the phenomena of science. Because the Rydberg constant is basic to the modern theory of the atom, it provides a connecting link between atomic constants and the accepted standards of measurement.

The value of the Rydberg constant in use today is based on the work of Drinkwater, Richardson, and Williams.² These observers measured the position of lines in the hydrogen spectrum against the primary standard wavelength of the red cadmium line. From the measured wavelengths of the transitions and the theory of the hydrogen atom, they calculated $R\infty$. This determination was modified by Cohen in 1952 in the light of increased knowledge.³ The result was accepted as an accurate value, although it differs significantly from the earlier results obtained independently by Houston⁴ and Chu,⁵ which were also adjusted by Cohen. As these two earlier experimenters measured lines in the hydrogen and helium spectra by using the 5016-Å line of helium as a reference, it was thought that the source of error might be in the wavelength of the helium line.

The Bureau's work shows that this was indeed the case. The helium 5016-Å line has been remeasured and the proper substitutions made in the calculations



A portion of an interferogram obtained with 20-mm interferometer spacing. Measurements were made in vacuum and wavelengths are given here with vacuum values. The 5016 line is shown with its measured value of 5017.0772 Å; to the left are two other helium lines; at right is the mercury-198 standard.

of Houston and Chu. The value obtained for $R\infty$ by combining all the modern determinations is $109737.312 \pm 0.008 \text{ cm}^{-1}$. This result is in excellent agreement with the presently accepted value ($109737.309 \pm 0.0012 \text{ cm}^{-1}$).

Measurements of the helium line at 5016 Å were made at the Bureau against two well-known wave-

W. C. Martin aligns the projecting lens of a system used in measurements to re-evaluate the Rydberg constant. This work involved measuring the wavelength of the 5016-Å helium line against a mercury standard. Liquid-nitrogen-cooled helium lamp is immersed in Dewar, lower right; mercury-198 lamp is at lower center.



lengths from a mercury-198 Meggers electrodeless lamp in an evacuated Fabry-Perot interferometer. The value for the line in question was determined as $5017.0772 \pm 0.0003 \text{ Å}$ in vacuum. To apply this wavelength to the data of Houston and Chu, the most recent dispersion formula for air was applied to the Bureau's data, yielding the value 5015.6782 Å in air. This increase from the value used by the earlier experimenters, which was Merrill's 1917 value of 5015.675 Å , is essentially confirmed by recent results obtained by Series and Field working at Oxford.⁶ They determined the wavelength as $5015.6775 \pm 0.0004 \text{ Å}$ in air. The weighted average of these two measurements ($5015.6779 \pm 0.0003 \text{ Å}$) replaced the earlier value accepted by Houston and Chu in the Bureau's calculations of the Rydberg constant.

In redetermining $R\infty$ from the data of Houston and Chu, wavelengths were corrected proportionally and applied in the usual calculations. The value obtained agrees very closely with the accepted value, providing an independent confirmation of the presently accepted $R\infty$ in the work of Houston and Chu. As the accuracy with which the Rydberg constant is known exceeds the accuracy of most other atomic constants with which it is connected, no further improvement is in demand at present.

¹ For further technical details, see *The value of the Rydberg constant*, by William C. Martin, *Phys. Rev.* (to be published); and *New wavelengths for some He I lines*, by William C. Martin, *J. Opt. Soc. Am.* (in press).

² Determinations of the Rydberg constants, e/m, and the fine structures of H_α and D_α by means of a reflexion echelon, by J. W. Drinkwater, O. Richardson, and W. E. Williams, *Proc. Roy. Soc.* **174**, 164 (1940).

³ The Rydberg constant and the atomic mass of the electron, by E. R. Cohen, *Phys. Rev.* **88**, 353 (1952).

⁴ A spectroscopic determination of e/m, by W. V. Houston, *Phys. Rev.* **30**, 608 (1927).

⁵ The fine structure of the line λ4686 of ionized helium, by D. Y. Chu, *Phys. Rev.* **55**, 175 (1939).

⁶ The wavelength of the helium line 5016 Å, by G. W. Series and J. C. Field, *Proc. of the Symposium on Interferometry*, National Physical Laboratory (June 1959).

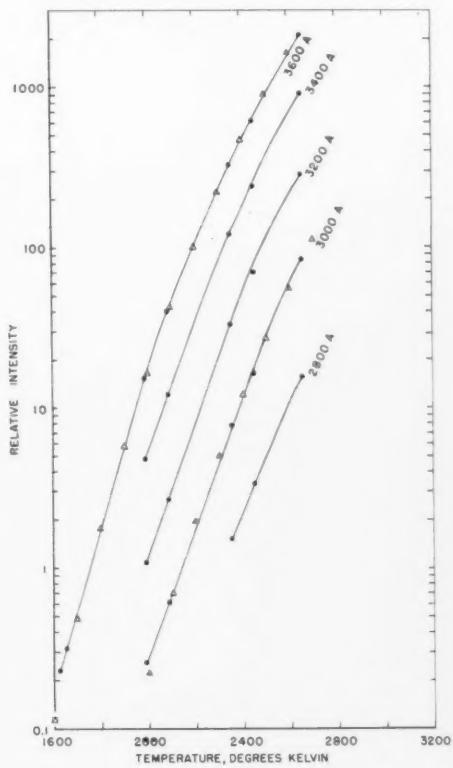
ABSOLUTE BASIS FOR ULTRAVIOLET RADIOMETRY

FOR a number of years, the Bureau has used a tungsten ribbon lamp as an approximate standard of spectral radiant energy in the near ultraviolet as well as in the visible and near infrared regions. This standard, instead of being based on an absolute measurement of spectral emission, is evaluated in terms of the temperature (based on pyrometric observations) and the emissivity of the tungsten ribbon. A more suitable standard would, of course, be a radiator approximating the ideal blackbody. In such a radiator, intensity is greater than that from any other body at the same temperature, radiations are essentially independent of the material of which the radiator is made, and variations in radiations with temperature and wavelength can be calculated from Planck's law. However, the development of such a standard has been difficult, pri-

marily because of the inability of materials to withstand the high temperatures necessary for the emission of measurable energy by a heated radiator in the ultraviolet spectral region.

R. Stair and R. G. Johnston of the radiometry laboratory have recently applied a number of developments in other fields to the construction of an improved graphite blackbody. The results of studies on materials for use at high temperatures, conducted by the Bureau's ceramic engineering group, were applied in constructing this blackbody, which shows potentialities as a practical standard of ultraviolet radiation. Among the advances that made this development possible is the industrial production of shielded radiofrequency generators which produce negligible interference in the operation of photoelectric amplifier systems. Equally important is the commercial production of high-purity graphite and of insulating materials with chemical and physical stability at high temperatures.

The blackbody standard of ultraviolet radiation constructed by the Bureau consists of a graphite core packed in boron nitride powder in a glass-fiber-wrapped porcelain container. R. G. Johnston inserts the blackbody into an induction coil operated by a high-frequency induction generator. The blackbody will be heated in an inert atmosphere.



Relative radiant-energy intensities at selected wavelengths as a function of blackbody temperature. Studies of the suitability of a newly developed blackbody for a standard of ultraviolet radiation show good agreement between observed data (solid circles) and calculations based on Planck's law (triangles).



Although experiments to determine optimum dimensions for the graphite blackbody are still underway, promising results have been obtained to date with a cylindrical enclosure 4½ in. long and 1½ in. in diameter, having walls $\frac{3}{16}$ in. thick. The exit end of the tube has a $\frac{3}{8}$ in. opening shielded by a conical graphite end-piece $\frac{3}{4}$ in. long, which contains a second similar opening.

The blackbody is heated by an induction method in a 6-turn water-cooled coil, powered by an industrial radio-frequency generator operating at 450 kc. The graphite tube is insulated by tightly packed boron powder in a high-temperature porcelain container. An alundum ceramic tube between the graphite core and porcelain container increases physical stability. By enclosing this "furnace" in an airtight helium-filled chamber, oxidation of the graphite at high temperatures is considerably reduced.

The distribution of radiant energy at various temperatures is detected by a double quartz prism spectro-radiometer. Two aluminized mirrors (one plane and the other spherical) are employed to focus an image of the blackbody opening on the slit of the optical system. The spectral energy output is detected by a photo-

multiplier, then amplified, and recorded on a strip chart.

Data were obtained with this arrangement for blackbody temperatures from 2,000° to above 2,600° K—measured with an optical pyrometer. Except at the highest temperatures, little depreciation of the graphite core occurred. Above 2,500° or 2,600° K, some chemical reaction between the heated graphite and boron nitride resulted in the production of boron carbide on the outer surface of the graphite. However, this does not appear to affect the radiation from the blackbody.

In comparing the radiation of this graphite enclosure at temperatures in the neighborhood of 2,000° K with calculations based on Planck's law, it was found that the radiation output closely approximates that of an ideal blackbody. Furthermore, calculations based upon the physical characteristics of the graphite tube support this conclusion. Such data indicate the practicability of using this rf-heated graphite blackbody as a reference standard for setting up working lamp standards of spectral radiant energy within the ultraviolet region to about 250 m μ . As a consequence, a group of strip lamps are being calibrated for absolute spectral radiant energy output through the region of 250 to 750 m μ .

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Section A. Physics and Chemistry, vol. 63A, No. 3, November-December 1959.

Multiphoton ionization of rare gases by electron impact. M. Krauss, R. M. Reese, and V. H. Dibeler.

Light scattering by commercial sugar solutions. C. J. Rieger and F. G. Carpenter.

Analysis of the first spectrum of ruthenium (Ru I), K. G. Kessler.

Supplementary Zeeman data for the first spectrum of ruthenium (Ru I), J. R. McNally, Jr. and K. G. Kessler.

Low even configurations in the first spectrum of ruthenium (Ru I), part 2, R. E. Trees.

Thermal degradation of polymers at high temperatures, S. L. Madorsky and S. Straus.

Influence of impurities on the pyrolysis of polyamides, S. Straus and L. A. Wall.

A preliminary list of levels and g-values for the first spectrum of thorium (Th I), R. Zalubas.

OH in the solar spectrum, C. E. Moore and H. P. Broida.

Use of Chebychev polynomials in thin film computations, K. D. Mielenz.

Section B. Mathematics and Mathematical Physics, vol. 63B, No. 2, October-December 1959.

Applications of a theorem on partitioned matrices, E. V. Haysworth.
Capacity requirement of a mail sorting device; II, A. J. Goldman.
Analytic comparison of suggested configurations for automatic mail sorting equipment, B. K. Bender and A. J. Goldman.
New method of solution for unretarded satellite orbits, J. P. Vinti.

Effect of sudden water release on the reservoir free outflow hydrograph, V. M. Yevdjevich.
Uniform asymptotic expansions for Weber parabolic cylinder functions of large orders, F. W. J. Olver.

Technical News Bulletin, Volume 43, No. 11, November 1959.
15 cents. Annual subscription \$1.50; 75 cents additional for foreign mailing.

Basic Radio Propagation Predictions for January 1960. Three months in advance. CRPL-D182. Issued October 1959. 15 cents. Annual subscription \$1.50; 50 cents additional for foreign mailing.

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Technical Notes are available only from the Office of Technical Services, U.S. Department of Commerce, Washington 25, D.C. (Order by PB number.)

Frequency dependence of VHF ionospheric scattering, J. C. Blair, Tech. Note 9 (PB151368) 75 cents.

Some applications of statistical sampling methods to outgoing letter mail characteristics, N. C. Severo, A. E. Newman, S. M. Young, and M. Zelen, Tech. Note 16 (PB151375) \$2.75.

Radio noise data for the International Geophysical Year July 1, 1957-December 31, 1958, W. Q. Crichtlow, C. A. Samson, R. T. Disney, and M. A. Jenkins, Tech. Note 18 (PB151377) \$2.50.

Variations of gamma cassiopeiae, S. R. Pottasch, Tech. Note 21 (PB151380) 75 cents.

Communication theory aspects of television bandwidth conservation, W. C. Coombs, Tech. Note 25 (PB151384) 50 cents.

Aerodynamic phenomena in stellar atmospheres—A Bibliography, Tech. Note 30 (PB151389) \$1.25.

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Atomic weights, E. Wichers, chapter 6. *Treatise on Analytical Chemistry, pt. 1. Theory and Practice, vol. I, section B. Application of Chemical Principles*, p. 161 (The Interscience Encyclopedia, Inc., New York, N.Y., 1959).

Concept and determination of pH, R. G. Bates, chapter 10. *Treatise on Analytical Chemistry, pt. 1. Theory and Practice, vol. I, section B. Application of Chemical Principles*, p. 361 (The Interscience Encyclopedia, Inc., New York, N.Y., 1959).

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TECHNICAL
NEWS
BULLETIN

U. S. DEPARTMENT OF COMMERCE

FREDERICK H. MUELLER, *Secretary*

NATIONAL BUREAU OF STANDARDS

A. V. ASTIN, *Director*

December 1959 Issued Monthly Vol. 43, No. 12

For sale by the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C. Subscription price, domestic \$1.50 a year; 75 cents additional for foreign mailing; single copy, 15 cents. Use of funds for printing this publication approved by the Director of the Bureau of the Budget (June 24, 1958).

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(The following U. S. Patents have recently been granted on NBS inventions and, except as noted, are assigned to the United States of America as represented by the Secretary of Commerce.)

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